A STUDY OF THE EFFECT OF TIME AND TEMPERATURE OF PRESOAKING AND THE TEMPERATURE OF ACETYLATION ON THE RATE AND DEGREE OF THE PARTIAL ACETYLATION OF DIFFERENT VARIETIES OF COTTON FIBERS

A THESIS

Presented to

the Faculty of the Graduate Division

by

Emmet Dennis Owens

In Partial Fulfillment

of the Requirement for the Degree

Master of Science in Textile Engineering

Georgia Institute of Technology

June 1953

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Approved:

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A STUDY OF THE EFFECT OF TIME AND TEMPERATURE OF PRESOAKING AND THE TEMPERATURE OF ACETYLATION ON THE RATE AND DEGREE OF THE PARTIAL ACETYLATION OF DIFFERENT VARIETIES OF COTTON FIBERS

ABSTRACT

It was the purpose of this problem to determine (1) the effect of time and temperature of presoaking on the rate and degree of the partial acetylation of different varieties of cotton fibers with the time and temperature of the acetylation held constant and (2) to study the effect of the acetylation temperature under constant conditions of presoaking.

The degradation of cotton fibers in the production of partially acetylated cotton is due primarily to the time and temperature used in the acetylation process. It is therefore of prime importance to determine some method of reducing the time and temperature of acetylation to a minimum and still obtain the desired acetyl content. The effect of the time and temperature of presoaking on the rate and degree of acetylation was thus determined in order to obtain the optimum conditions of presoaking.

Six different varieties of cotton fibers representing a wide range of physical properties were selected for this study. The cottons selected were Memphis, Empire Bale 92, Bob Shaw, Stoneville 2B, Acala 1517 and Lockett 140.

All acetylations in Part 1 of this study were carried out at 64° F. for 45 minutes. The time of presoaking was varied from 10 minutes to 240 minutes and the temperatures used were 70° F., 100° F., 130° F., and 170° F. In Part 2 all acetylations were carried out for 45 minutes at different temperatures ranging from 58° F. to 82° F. in six degree increments. The optimum presoaking conditions, 10 minutes at 170° F., were determined from Part 1 and used for all acetylations in Part 2.

All presoakings and acetylations were carried out in a one-pound package dyeing machine modified with cooling coils in such a manner that positive temperature control was attained at all times.

The acetylated samples were analyzed for acetyl content, moisture per cent, fiber fineness, fiber strength, and evenness of acetylation. The values of the acetyl contents were plotted against the times and temperatures of presoaking and the temperatures of acetylation and the graphs carefully analyzed.

From the results obtained it was concluded that an increase in the temperature of the glacial acetic acid used for presoaking increases the rate in which maximum acetylation is reached but it does not effect the maximum degree of acetylation obtained under constant acetylation conditions. It was found that different fibers acetylated at different rates and degrees and generally the more immature the fiber the greater the rate and degree of acetylation and conversely the more mature the fiber the less the rate and degree of acetylation under constant conditions of presoaking and acetylation. Finally, it was determined that

the higher the temperature used in acetylation, the greater the degree of acetylation and the greater the degradation of the cotton fibers.

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CHAPTER I

INTRODUCTION

In recent years considerable attention has been focused on the use of partially acetylated cotton as a textile fiber. The chemical modification of cotton by partial acetylation imparts many new and valuable properties to the cotton fiber while at the same time its physical appearance and general textile qualities remain virtually unchanged. The new characteristics of the chemically modified cotton fibers include resistance to mildew and microbiological rotting; resistance to the effects of high temperature and continued heat; low moisture regain and high electrical resistance, as compared with untreated cotton; and resistance to coloring with many direct dyes.[1]

Partially acetylated cotton appears to be unaltered physically both in the acetylating mixture and in the dried condition. It possess the same tensile strength per unit weight of cellulose and the same elasticity as unacetylated cotton, and shows only a somewhat harder handle. The characteristic structure of the cotton fiber is preserved entirely; even the light reflections in polarized light are the same. The only noticeable difference is that low acetylated fibers appear to

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[1] Albert S. Cooper, and others, "Partial Acetylation of Cotton," Textile Industries, 116-1 (January, 1952), 97-102, 194-95.

be more rod-like than the original fibers and the fibers increased in weight by approximately 25 per cent without shrinkage in length.[2]

The degree of acetylation of the cotton fiber considered in this study is based on that of the monacetate, corresponding to approximately one-third of complete acetylation or about 21 per cent acetyl content. The partial acetylation of cotton is an example of esterification which consists in the introduction of acetyl groups throughout the cellulose to replace an equivalent part of its hydroxyl groups of which cotton contains about 30 per cent by weight.

History.--In 1901 two British chemists, C. F. Cross and E. J. Bevin, produced a low acetylated cotton fiber by treating bleached cotton with boiling acetic anhydride in the presence of distilled sodium acetate. Although possessing the properties of partially acetylated cotton; i.e. (a) it absorbed much less than the usual amount of hydroscopic moisture from the atmosphere and (b) it was uneffected by the usual solvents for cellulose acetates; these fibrous acetates were of no practical use since they had lost all tensile strength, probably due to the high reaction temperature.[3]

C. F. Cross and Briggs altered the original process in 1907 with a view to conducting the acetylation at low temperatures. In this process the cellulose was impregnated with a mixture of acetic anhydride, acetic acid, and a salt like condensing agent and then squeezed until a certain

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[2]E. Chippindale, "Low Acetylation of Cellulose Fibers," Society of Dyers and Colourists, 1 (1934), 142-49.

[3]Ibid., p. 142.

increase in weight was obtained and afterwards acetylating by warming in a closed apparatus. This process was found to be of no commercial value due to the complicated apparatus required and to the fact that irregularly acetylated fibers were obtained due to lack of control during the reaction.[4]

Work continued on the partial acetylation of cotton, mainly in England, with the object of producing partially acetylated cotton which would retain its tensile strength after acetylation. In 1928 partially acetylated cotton yarns were the subject of E. P. 280,493 and were registered under the names of Cotopa (non-lustrous) and Crestol (lustrous).[5]

The usual acetylation procedure today is essentially the one that is described in A. C. Thaysen's British patents and intended particularly to impart resistance to mildew and rot.[6]

Extensive investigation has been carried out on partially acetylated cotton in order to determine more about the new properties imparted to the cotton fiber. The Department of Agriculture, through its Southern Regional Research Laboratory, New Orleans, La., has conducted extensive research in the production and uses of acetylated yarn and fabrics.

In 1920, C. Doree reported that cotton fabrics, acetylated to the monoacetate stage, immersed in sea water were completely uneffected after

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[4]Ibid., p. 142.

[5]Ibid., p. 145.

[6]Charles Goldthwait, "Acetylation to Make a New Textile," Yearbook of Agriculture, 1950-51, p. 422.

sixteen weeks while unacetylated cotton was destroyed in three weeks.[7] Thaysen and his coworkers found that partially acetylated cotton resisted rotting by soil micro-organisms when buried in the ground and reported on a partially acetylated cotton fabric which withstood such burial for seven years.[8]

Partially acetylated cotton has an advantage over goods treated with the usual mildew and rot-proofing agents because it is not discolored, odorous, sticky, nor poisonous.[9] It has been found that bags made of partially acetylated cotton for holding zeolite in household water-softening systems remained intact during a normal years service, whereas ordinary bags developed holes in a month or two. Acetylated fish net twines tested in ocean water were found to last two to three times as long as twines treated with tar and eight to ten times as long as untreated twines. The nets of acetylated twines are more flexible and easier to handle than nets protected by tar.[10] Tests on acetylated sandbags showed that after two years exposure to the weather the bags on the bottom of the pile retained 35 to 40 per cent of their breaking strength and those on the top retained 35 to 100 per cent of their breaking strength in contrast to complete deterioration of untreated bags in one-fourth the time.[11]

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[7] C. Doree, "The Action of Sea Water on Cotton and Other Textile Fibers," Biochemistry Journal, 14 (1920), p. 713.

[8] G. F. Goldthwait, J. McLaren and S. T. Voorhies, Jr., "Acetylated Cotton Highly Resistant to Rotting," Textile World, 96-1 (1946), p. 117.

[9] Ibid., p. 116

[10] Copper and others, op. cit., p. 98.

[11] Walter Scott, "Some Recent Developments in Cotton Research at Southern Regional Research Laboratory, Textile Research Journal, 19 (1949), p. 440.

The resistance of partially acetylated cotton to high temperatures and continued heat have been extensively investigated. Tests were made on the comparison between acetylated cotton fabrics and unacetylated fabrics at 160° F. under different conditions of humidity, etc. and it was found that in all cases the acetylated fabrics were much more heat resistant. The tensile strength of the unacetylated cotton fabrics were greatly reduced while that of the acetylated cotton were only slightly affected.[12] A sheeting of partially acetylated cotton has been found to last four to five times as long as the usual cotton canvas covers for laundry "hot head presses". Examples of the effect of dry heat with full exposure to air in an ordinary drying oven indicated that the acetylated cotton would require more than seven times as long to become weakened to half strength at 320° F.[13]

Acetylated cotton has been used as tire cords with good results by the British for a number of years. It was found to be particularly effective in tropic climates.[14] Acetylated cotton swells less when wet and it is of particular value when cotton textiles have to maintain their weight, length and width unchanged under varying weather conditions. Partially acetylated cotton is also said to bond better than ordinary

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[12] E. Honold, J. Poynot and A. Cuculla, "Heat Resistance of Partially Acetylated Cotton Fabrics," Textile Research Journal, 22-1 (1952), 25-29.

[13] A. S. Cooper and others, op. cit., p. 97.

[14] C. M. Blow and W. J. Knight, "Textile Fibres and Rubber as Associated Materials for Manufacture," Textile Institute Journal Proceedings, 35 (1944), p. 12.

cotton with plastics, such as cellulose acetates, etc. Owing to its low hygroscopicity, about half of that of unacetylated cotton, it finds use as electrical insulation material.[15] As more fundamental knowledge concerning partially acetylated cotton is discovered the uses of this cotton will greatly increase.

Statement of the Problem.--The partial acetylation of cotton fibers is carried out in three principal steps: (1) preparation of the cotton for acetylation, (2) the acetylation reaction and (3) washing and drying.

It was the purpose of this study to determine (1) the effect of the time and temperature of presoaking on the rate and degree of acetylation of different varieties of cotton fibers with the time and temperature of the acetylation held constant and (2) to study the effect of the acetylation temperature on the degree of acetylation under constant conditions of presoaking.

Importance of the Study.--At the present time partially acetylated cotton is being produced only on a limited scale in this country and although considerable research is being conducted, primarily by the Southern Regional Research Laboratory, New Orleans, La., there still remain many inherent fundamental physical and chemical aspects of the acetylation reaction to be determined. Relatively little research has been carried out on the raw stock itself and it is the purpose of this study to determine the relationship of the presoaking conditions, using glacial

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[15] C. F. Goldthwait, op. cit., p. 421.

acetic acid as the presoaking reagent, to the rate and degree of acetylation. Previous acetylations of raw stock have usually been carried out using an overnight presoak at room temperatures. Acetylated cotton produced on a commercial scale must necessarily be a continuous process carried out within the shortest possible time with the least amount of damage to the fibers. Heuser[16] has shown that the amount of degradation of cellulose depends upon the time and temperature of acetylation, independently of the acetyl values reached. It is therefore of prime importance to determine some method by which the time and temperature of acetylation could be reduced and still obtain the desired acetyl values. To that end, various conditions of time and temperature of presoaking and temperatures of acetylation were studied, the acetyl content determined and the results analyzed.

Method of Approach.--Six different varieties of raw cotton were selected for this study. These cottons were chosen to represent a wide range of physical properties such a maturity, fiber fineness and tensile strength as illustrated in Table 1.

In the first part of this study the cottons were acetylated under uniform conditions of time and temperature, i.e. 45 minutes at 64° F., with the time and temperature of presoaking as variables. The times used were 10, 30, 60, 120, and 240 minutes and the temperatures selected were 70° F., 100° F., 130° F., and 170° F.

In the second part of this study the time and temperature of presoaking was held constant at the optimum conditions of presoaking as

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[16] Emil Heuser and others, "Acetylation of Cellulose in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40 (1948), 1500-06.

determined from Part I. These conditions were determined to be 10 minutes at 170° F. The acetylation temperature was varied with the time of acetylation being held constant at 45 minutes. The temperatures used in the acetylations were 58° F., 64° F., 70° F., 76° F., and 82° F.

All acetylations and presoakings were carried out in a one-pound package dyeing machine modified in such a manner that the above temperatures were easily obtainable and held constant.

The acetylated cotton was analyzed for acetyl content, fiber fineness, tensile strength and uniformity of acetylation. The values of the acetyl content were plotted against the times and temperatures of presoaking and the temperatures of acetylation. These graphs were analyzed to determine the effect of time and temperature of presoaking on the rate and degree of acetylation and the effect of the temperature of acetylation on the degree of acetylation of the different varieties of cotton fibers.

Table 1. Summary of Physical and Chemical Properties of the Original Cotton Fibers

Cotton	% Moisture Content	Micro-naire Mg/inch	Pressley Index	Angle 40%*	Maturity(%) NaOH Method*
Memphis	5.8	2.45	7.15	37.92	38
Empire Bale 92 Bale 249290	5.8	3.70	7.30	34.08	72
Bob Shaw	5.4	5.10	8.42	31.32	88**
Stoneville 2B	5.9	3.20	8.07	31.20	80
Acala 1517	6.0	4.00	8.93	29.76	86
Lockett 140	5.9	5.67	7.51	36.18	92

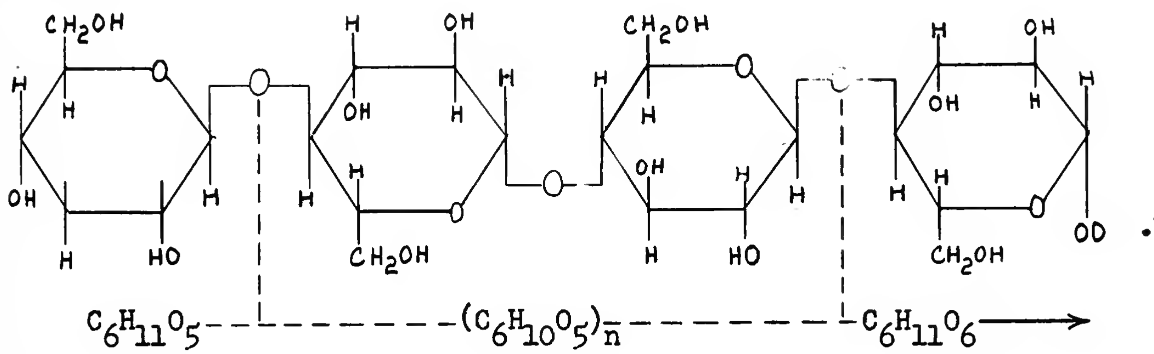
*These results were furnished by the Southern Regional Research Laboratory, New Orleans, La.

**Maturity was determined by the Areatometer.

CHAPTER II

THEORETICAL CONSIDERATIONS

The structure of the cellulose molecule may be represented as follows:[1]



The glucose units, $C_6H_7O_2(OH)_3$, of which the cellulose chain is composed, contains three hydroxyl groups which are free to react to form cellulose derivatives. The acetylation of cellulose is an example of esterification and consists of replacing the hydroxyl groups by acetyl groups. The number of hydroxyl groups replaced in a particular reaction will determine the degree of modification of the cellulose and the attendant changes in properties.[2]

The acetylation of cotton fibers with glacial acetic acid alone occurs very slowly, even under the effects of high temperatures. Malm

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[1]Petrus H. Hermans, Physics and Chemistry of Cellulose Fibres. New York: The Elsevier Publishing Co., Inc., 1949, p. 8.

[2]Charles F. Goldthwait, "Acetylation to Make a New Textile," Yearbook of Agriculture, 1950-51, p. 421.

and Clark[3] boiled surgical cotton in glacial acetic acid and at the end of 96 hours only 6.2% acetyl content was reached. Even after 500 hours only 6.4% acetyl content was obtained and that with considerable degradation to the fibers. In sealed glass tubes at 140°- 145° C., 7.5% acetyl content was obtained after 48 hours. Again the long time and high temperature led to considerable degradation. The use of acetic anhydride in place of glacial acetic acid results in greatly increased acetyl content, 38.26% at 118° C., but again the cellulose is considerably degraded.[4] Heuser and his coworkers[5] have shown that the degradation of cellulose depends upon the time and temperature of acetylation independently of acetyl values reached.

Cellulose acetate thus cannot be prepared by direct esterfication with glacial acetic acid, instead it is necessary to use acetic anhydride. The reaction may be expressed by either of the following equations:[6]

$$(1)\quad C_6H_7O_2(OH)_3 + 3(CH_3CO)_2O \longrightarrow C_6H_7O_2(OCOCH_3)_3 + 3CH_3COOH$$

$$(2)\quad C_6H_7O_2(OH) + 3(CH_3CO)_2O \longrightarrow 2C_6H_7O_2(OCOCH_3)_3 + 3H_2O$$

The theoretical anhydride requirement in the first case is 189 per cent, whereas in the second case it is only one-half this amount. Since a con-

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[3]C. J. Malm and H. T. Clark, "The Action of Fatty Acids on Cellulose," Journal of the American Chemical Society, 51 (January, 1929), pp. 274-77.

[4]Emil Heuser, The Chemistry of Cellulose. New York: John Wiley and Sons, Inc., 1947, pp. 227-28.

[5]Emil Heuser and others, "Acetylation of Cellulose in Phosphoric Acid Solution," Industrial and Engineering Chemistry, 40 (1948), 1500-06.

[6]Emil Heuser, op. cit., p. 230.

siderable excess of 189 per cent is actually required for complete substitution, it is probable that the reaction is more truely expressed by the first equation although it is difficult to decide just which reaction is more likely.

The acetylation reaction is greatly facilitated by the use of certain catalysts, such as sulphuric and perchloric acids, which are actually dehydrating agents although they do not seem to act as such when acetic anhydride is used for acetylation. The function of the catalyst is not clearly understood. One theory advanced in the case of sulfuric acid is that the acid reacts with the acetic anhydride to form acetylsulfuric acid which functions as the acetylating agent and regenerates sulphuric acid to form a complete cycle. The chief function of the sulfuric acid seems to be as an aid in the swelling of the cellulose material and to degrade it.[7]

In this study perchloric acid, which is the most powerful catalyst for the acetylation of cellulose,[8] was used. In acetic acid/acetic anhydride systems, strong acids such as perchloric acid exhibit "superacidity" which may be accounted for by postulating the existence of highly acidic ions and in this respect perchloric acid is stronger than sulfuric acid.[9] In acetylation, perchloric acid first forms an addition complex with the cotton; this is slowly broken down by the introduction of acetyl

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[7]Emil Heuser, op. cit. pp. 228-232.

[8]Emil Heuser, op. cit. p. 240.

[9]H. A. E. Mackenzie, "Acetic Acid/Acetic Anhydride System: Properties," Transactions of the Faraday Society, 44 (1948), 159-71.

groups and at the same time small amounts of perchloric ester groups are formed. Theoretically the triacetate would thus contain no perchloric acid.[10]

The amount of catalyst used effects the rate of acetylation, thus with an increase in the amount of catalyst, the rate of acetylation increases.[11] However the amount of absorbed catalyst is not as important as the uniformity of its distribution in the cellulose. High reactivity of cellulose towards acetylation is obtained where there is a rapid and uniform absorption of the catalyst.[12]

The rate of acetylation is greatly influenced by the moisture content of the cellulosic material. The rate of reaction increases with increasing moisture content until the maximum is reached at about 25 to 30 per cent. The increasing water content causes the capillaries of the fibers to enlarge which favors the diffusion of the acetylating mixture into the inner parts of the fiber. When the moisture content reaches 25 to 30 per cent the fibers have reached saturation, i.e. all capillaries are filled with water, so that further increase of water content has no effect upon the diffusion and hence upon the rate of reaction.[13] Any

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[10] Genevieve Petitpas, "The Perchloric Acid Catalyst of the Acetylation of Cellulose," Mem. services chim etat., 31 (1944), 178-86, as abstracted in Chemical Abstracts, 40-2 (1946), 7613.

[11] Emil Heuser, op. cit., p. 231.

[12] C. J. Malm and others, "Treatment of Cellulose Prior to Acetylation," Industrial and Engineering Chemistry, 44 (1952), 2904-09.

[13] Emil Heuser, op. cit., pp. 243-45.

compound which enlarges the lattice spacings and permits faster diffusion into the crystallite increases the reaction rate.[14]

Presoaking with glacial acetic acid has the same general effect as presoaking in water. The use of glacial acetic acid is preferred in the partial acetylation of cotton as water must be completely removed prior to acetylation. Pretreatment with glacial acetic acid or water shorten the time necessary to reach a certain acetyl content and thus the degradation of the cellulose is reduced and the molecular weights of the acetates are higher.

Presoaking in water increases the strength of the cellulose acetate, the maximum strength being attained with 20 per cent moisture content. Previous swelling in glacial acetic acid however, has no effect on the strength of the cellulose.[15]

The effect of time and temperature of presoaking with glacial acetic acid was determined in this study.

The acetylation of cellulose is a heterogenous reaction which is due both to fiber and micellar structure. X-ray studies have shown that in the acetylation of cellulose the reagent penetrates the fiber, attacking first the surface of the micelle which is changed into the triacetate and then proceeds inwards, producing between the unreacted interior and the completely reacted surface a partially reacted area in which only

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[14] W. A. Sisson, "X-Ray Diffraction Behavior of Cellulose Derivatives," Industrial and Engineering Chemistry, 30 (May, 1938), p. 532.

[15] E. Elod, H. Schmid-Bielenberg and I. Thoria, "Acetylation of Cellulose Fibres," Angew Chemistry, 47 (1934), 465-68, as abstracted in Journal of the Textile Institute, 25 (1934), A420.

one or two hydroxyl groups have been replaced.[16] In the crystalline structure where the cellulose chains are densely packed and regularly oriented the reaction is slow. In the amorphous portion of the fiber, where access of the reagents is greater due to the expanded structure, the reaction is much faster. No definite border exists between these regions but transitional regions exist in an intermediate degree.[17] Thus the amorphous regions are accessible for chemical reactions as well as the surface of the crystalline regions.[18] X-ray studies indicate the crystalline fraction of cotton to be 70 per cent within small experimental error.[19]

Sisson[20] states that the kinetics of cellulose derivative formation are determined largely by the velocity of diffusion rather than by the speed of reaction. The rate in the first stage is thus determined by the nature of the intermicellar spaces through which the reagent becomes accessible to the micellar surfaces. In the second and final phase, the reaction progresses from the surface of the micelle to the interior. Here the rate depends on the velocity of diffusion of the reagent through the reacted surface layer to the unattacked center of the micelle with

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[16] W. A. Sisson, _op. cit._, p. 531.

[17] Alexander Meller, "Reactivity of Fibrous Cellulose," _Journal of Polymer Science_, 4 (1949), 619-28.

[18] L. Ward, "Crystallinity of Cellulose and Its Significance for the Fiber Properties," _Textile Research Journal_, 20 (1950), 363-372.

[19] Petrus Hermans, _op. cit._, p. 316.

[20] W. A. Sisson, _op. cit._, p. 531.

the reaction taking place at the boundary surface between these two portions. At this stage, the rate of reaction depends primarily on the permeability of the reacted layer to the reactant molecules.

The reactions of cellulose according to Heuser[21] may be divided into four possibilities which are derived from the physical and chemical reactivities of the hydroxyl groups. First, the reagent displays an exclusive preference for one of the two types of hydroxyl groups and the reaction proceeds in a topochemical fashion. A break in the rate of reaction occurs when all of the reactive and easily accessible hydroxyl groups have reacted and the reagent begins to penetrate the denser crystalline regions. Second, the reaction displays a greater preference for one of the two types of hydroxyl groups than the other. The reaction proceeds in a permutoid fashion, i.e. all of the preferred hydroxyl groups react faster than the less preferred regardless of where they are located. A break in the reaction occurs when the reaction of the chemically preferred hydroxyl groups approaches completion and the other reaction of the less preferred groups becomes more pronounced. Third, the reagent exerts no preference for any of the hydroxyl groups. A break in the reaction occurs when all the hydroxyl groups located in the amorphous region have reacted and the reagent begins to enter the crystalline regions. Fourth, the reagent shows no preference either chemically for any of the hydroxyl groups or physically for amorphous regions. The reagent overcomes the barriers of the microscopic and submicroscopic structures and reacts quickly with

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[21] Emil Heuser, "Factors which Influence the Kinetics of Cellulose Reactions," Textile Research Journal, 20 (December, 1950), pp. 363-72.

all three hydroxyl groups. The process of acetylation seems to follow the third possibility, however the actual reaction may possibly be a combination of any two or more of the above possibilities.

Cellulose reactions do not seem to follow a single pattern, the type of reaction varies depending on its reaction partner, its concentration, the reaction medium, temperature, etc. The exact pattern that the acetylation reaction follows is difficult to determine.

CHAPTER III

INSTRUMENTATION AND EQUIPMENT

Morton one-pound Package Dyeing Machine modified with cooling coils for temperature control (See Figs. I and II)

Brabender Moisture Tester

Sheffield Micronaire, Model No. 60600

Pressley Fiber Strength Tester

Forced Circulation, electrically heated Oven

Laboratory Cutting Mill, Willey Intermediate Model

Precision Balance

Kodak Timer

Glacial Acetic Acid

Acetic Anhydride

Perchloric Acid, 60%

Ammonium Hydroxide, U.S.P., 28%

Hydrochloric Acid, C.P.

Sodium Hydroxide, C.P.

Methyl Alcohol, Anhydrous, Acetone Free

Sodium Sulfate, Anhydrous

Triton X-100 (Wetting Agent)

Dyestuffs
    Chlorantine Fast Blue 3RRL (Ciba)
    Celliton Fast Yellow RRA (Gen. Dyestuff Corp.)

Other standard laboratory equipment and materials

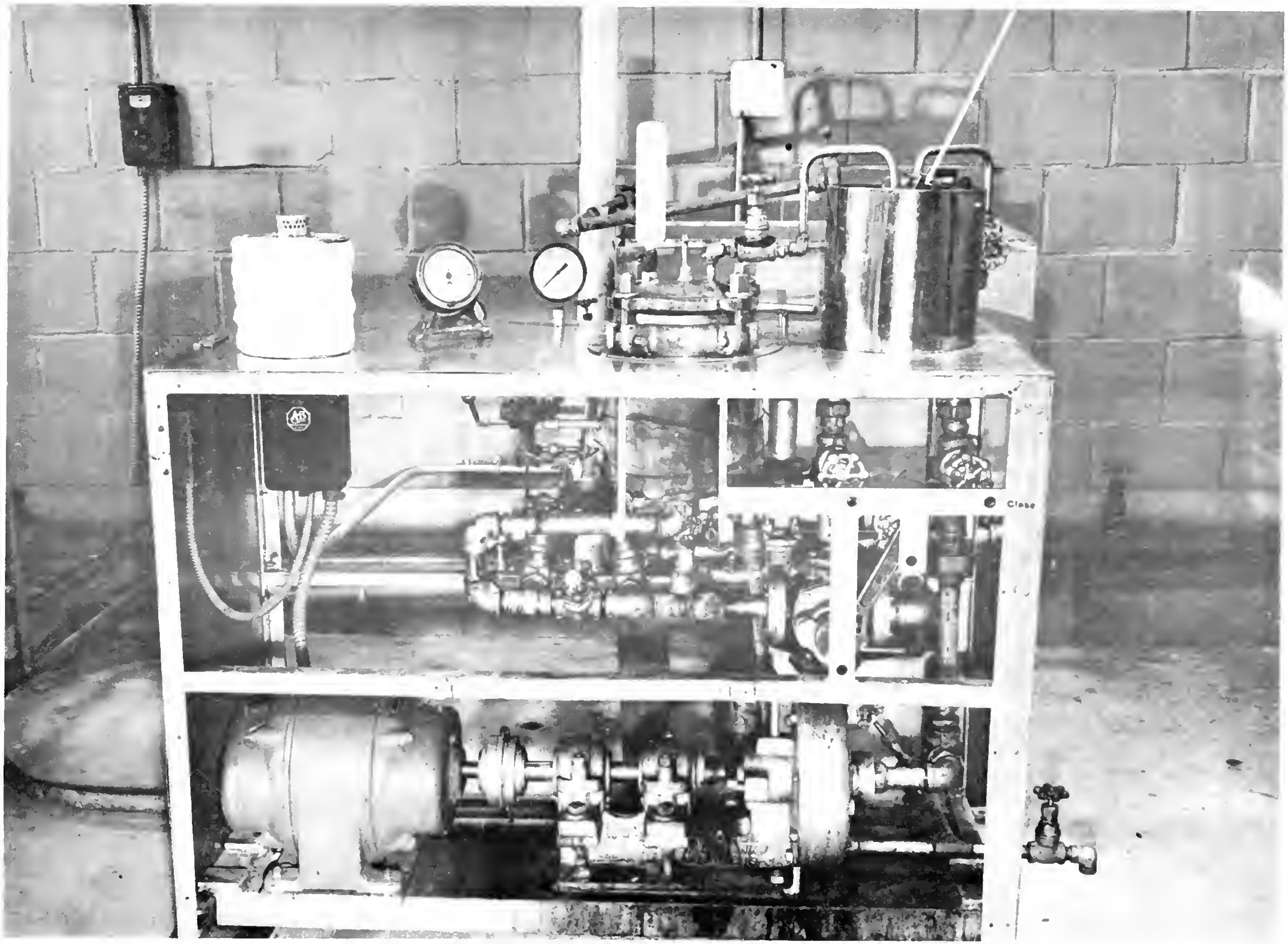

Figure 1. Front View of the Acetylation Machine.

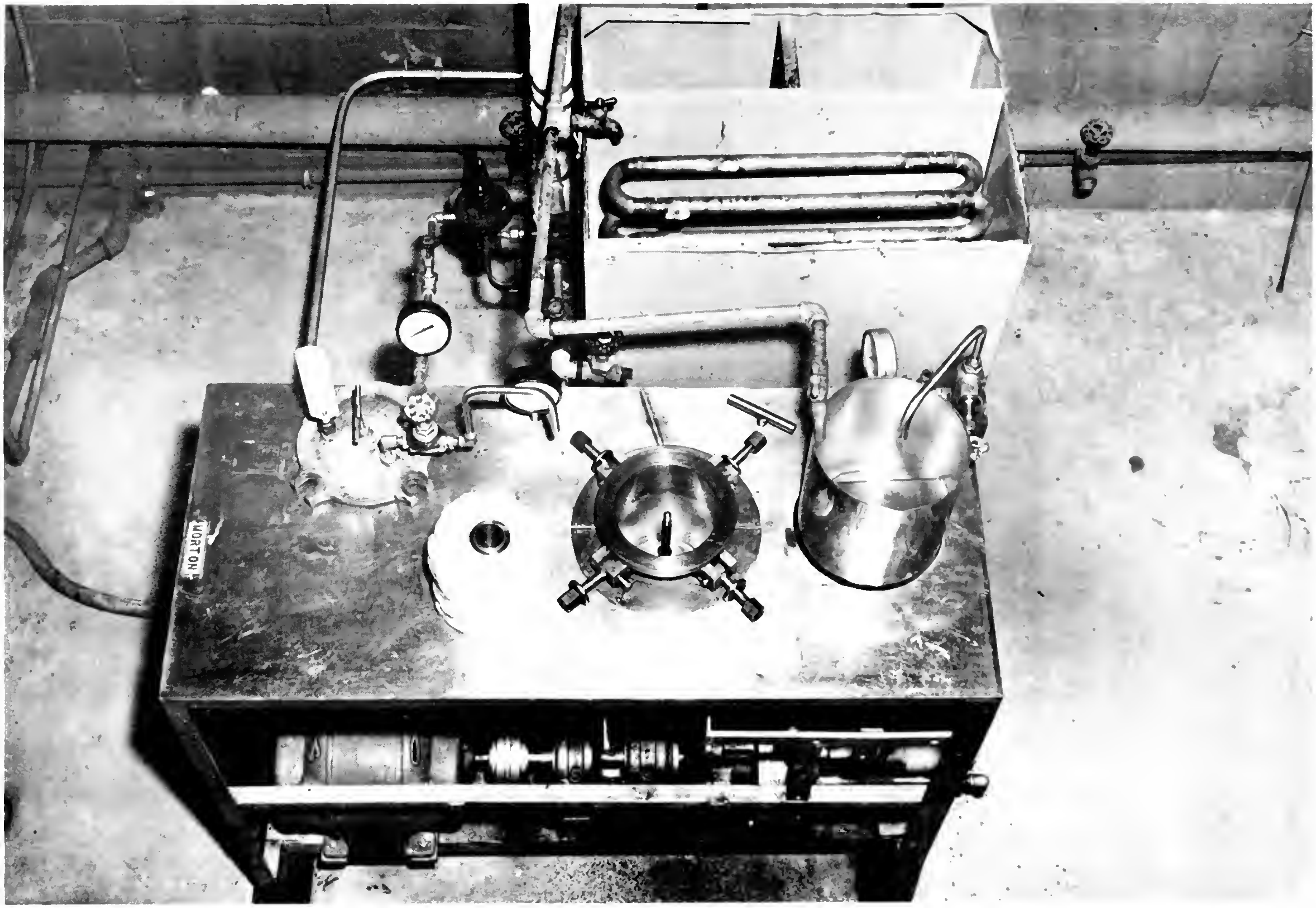

Figure 2. Top View of the Acetylation Machine.

CHAPTER IV

EXPERIMENTAL PROCEDURE

Selection of Cotton Fibers

Six varieties of cotton fibers were selected for this study. These cottons were selected on the basis of possessing a wide range of variables such as maturity, fiber fineness and tensile strength. The cottons selected were Memphis, Empire Bale, Bob Shaw 92, Stoneville 2B, Acala 1517, and Lockett 140.

Testing Prior to Acetylation

Moisture Content - Brabender.--The per cent moisture contained in the cotton was determined using the Brabender Moisture Tester. The cotton was conditioned for at least 48 hours at 70° F. and 65% relative humidity before being tested. The moisture tester was preheated for at least 15 minutes at 110° F. before being used. The fiber cans were placed in the machine prior to preheating. At the completion of the warm up period the perforated cans were removed from the machine and ten gram samples of conditioned cotton carefully weighed in the cans using the balance provided with the tester. The cans were then placed back into the oven of the tester and readings taken until equilibrium was established. The per cent moisture is read directly from the calibrated scale on the machine.

Fiber Fineness - Micronaire.--The fiber fineness was determined using the Sheffield Micronaire, Model No. 60600. The instrument was operated in accordance with the instruction phamplet published by the manufacture.[1]

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[1] Instructions For the Use of the Sheffield Micronaire, Dayton, Ohio: The Sheffield Corporation.

Fiber Strength - Pressley.--The cottons were tested for fiber strength in accordance with the method recommended by the A.S.T.M.[2]

Preparation of the Cotton Prior to Acetylation

Conditioning.--Prior to acetylation the cotton fibers were conditioned for at least 48 hours at 70° F. and 65 per cent relative humidity. After conditioning the required length of time the cotton was carefully weighed and placed in large manila envelopes until required for acetylation.

Presoaking.--A one-pound package dyeing machine modified with cooling coils was used for presoaking the cotton prior to acetylation. This modification consisted of adding cooling coils in series with the pipes carrying the liquid to the reaction chamber. Valves were placed in the pipe lines so that the cooling coils could be cut off from the main lines if desired. The cooling coils were placed in a large reservoir or tank in which ice and/or water could be placed. The machine was thoroughly rinsed out with used acetylation mixture prior to adding the conditioned cotton in order to be certain that the machine was free of water. The cotton to be acetylated was placed on a stainless steel spindle and stainless steel screens were placed between each variety of cotton. Six 25 gram samples plus 90 grams of filler cotton or twelve 20 gram samples were placed in the machine at a time. The total weight of cotton used was kept constant at 240 grams. Sixteen liters of glacial acetic acid was added after the cotton was placed in the machine. The

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[2]A.S.T.M. "Tentative General Methods of Testing Cotton Fibers (No. D414-47T)," Standards on Textile Materials, (October, 1948), p. 229.

valves were so adjusted that the cooling coils were filled with acid and could be used if so desired. The acid was adjusted to flow inside-out. The reacting chamber was steam jacketed and after the machine was filled with acid the steam (40 pounds pressure) was turned on and the acid quickly brought up to the desired presoaking temperature. The thermometer on the acetylating chamber cover was used in determining the presoaking temperature. When the desired time of presoaking was reached, the presoaking acid was quickly cooled to 80° F. through the use of the cooling coils. The cooling coils were packed with ice and water just prior to the end of the presoaking period. When the temperature of the presoaking solution reached 80° F., it was quickly drained from the machine into a five gallon acid carboy and set aside to be used for the next acetylation mixture.

Acetylation

Preparation of the Acetylation Mixture.--The acetylation mixture was composed of three parts of glacial acetic acid, one part of acetic anhydride and 0.15% of perchloric acid based on the total volume of acid and anhydride. The total volume of acetylation mixture used in the machine consisted of 12.6 liters of glacial acetic acid, 4.2 liters of acetic anhydride and 25.5 cc. of perchloric acid. The glacial acetic acid previously used for presoaking was used for acetylation. At least 12 hours prior to acetylation the acetic anhydride was added to 11.6 liters of acetic acid and the mixture allowed to stand in order that any water in the acid would be converted into acetic acid by reaction with the anhydride. The final acetylation mixture was made up just prior to the acetylation.

The acid-anhydride solution was cooled to 50° F. before adding the catalyst. The perchloric acid was mixed with the remaining liter of acetic acid and slowly added, with agitation, to the cooled acid-anhydride solution. There was an initial temperature rise of about 4°-8° F. when the perchloric acid was added after which mixture was again cooled to 50° F.

Acetylation.--Upon completion of presoaking and after draining the machine of the acid, the acetylating mixture was added to the machine through the reserve tank. The valve to the cooling coils was opened and the acetylating solution was pumped through the coils. The valves to the cooling coils were manipulated in such a manner that the temperature was held constant during the acetylation. All acetylations were carried out for 45 minutes; at the end of which time the acetylation mixture was quickly drained and the cotton thoroughly washed. In part I of this study the temperature of acetylation was held constant at 64° F. and in part II the temperature was varied in six degree increments from 58° F. to 82° F. for the different acetylations.

Treatment After Acetylation

Washing.--After the machine was drained the samples were washed with cold water. The washing was continued for 30 minutes after which the machine was drained. The machine was again filled with water and 50 cc. of concentrated ammonium hydroxide added and circulated through the cotton for about ten minutes or until tests with litmus paper indicated the wash water was basic. The machine was again drained and cold water

circulated with the drain valve opened until tests with litmus paper indicated the water passing out of the machine was neutral. The machine was then drained and the cotton removed.

Drying.--The excess water was removed from the acetylated cotton by the use of a hydro-extractor. The samples were then dried in hot circulating air at 180° F.

Conditioning.--Upon completion of drying the acetylated cotton was conditioned at 70° F., and 65 per cent relative humidity for at least 48 hours prior to making any analysis.

Testing After Acetylation

Physical Properties.--The moisture content, fiber fineness and fiber strength of the acetylated cottons were determined using the procedures previously described.

Analysis for Acetyl Content.--Two to three grams of the conditioned samples of acetylated cotton were carefully handpicked from the original 20 or 25 gram samples and ground up in the Wiley Mill. They were then transferred into tared 250 ml. Erlenmeyer flasks. The flasks were again accurately weighed and the exact weight of the samples calculated. From the per cent moisture previously determined, the dry weight of the samples were calculated. Fifty milliliters of 75% methyl alcohol were added to each sample and heated for 15-20 minutes at 140° F. Fifty milliliters of approximately 0.5 N sodium hydroxide solution was then added to each flask and the flasks again heated to 140° F. and allowed to stand 12 to 24 hours before

titrating. Two blanks of unacetylated cotton were included with each set of samples and carried through the same procedure. Two to three drops of phenolphthalein were added to the flasks as an indicator and the saponified samples then titrated with approximately 0.5 N hydrochloric acid until the pink color disappeared. If the pink color reappeared on standing more acid was used until the solution was completely neutralized. The percentage of acetyl content was then calculated according to the following equation:

$$\text{Per cent acetyl content} = \frac{(\text{cc. acid used by blank}) - (\text{cc. acid used by sample})}{\text{Dry weight of the sample}} \times 4.302 \times N$$

where 4.302 is the milliequivalent weight of the acetyl group times 100 and N is the normality of the hydrochloric acid.

Dye Test for Evenness of Acetylation.--The uniformity of acetylation is determined qualitatively by a dyeing test. The dried samples (or portions thereof) were introduced in a bath of 70 to 1 liquor ratio containing 4% Chlorantine Fast Blue 3RRL, 4% Celleton Fast Yellow RRA and 5% Triton X-100 calculated on the weight of the sample. After dyeing for 30 minutes at 180°-190° F., 50% of anhydrous sodium sulphate (on the weight of the samples) was added and the dyeing continued for 30 minutes more. The samples were then removed from the dye bath and washed well with cold water and then dried in the oven at 210°-212° F. Unacetylated cotton was dyed blue while acetylated cotton was dyed yellow. From the results of this test the evenness of the acetylation could be readily observed.

CHAPTER V

DISCUSSION OF RESULTS

In Part I of this study, the effect of time and temperature of presoaking on the rate and degree of acetylation was determined. The acetyl contents obtained at different times and temperatures are shown in Tables 4 through 7 and Figures 3 through 6.

A study of this data indicated that the temperature of the presoaking acid had a very marked effect of the degree of acetylation. Thus it can be seen that as the temperature was increased, particularly at the shorter presoaking periods, the degree of acetylation rapidly increased. This was especially true in the case of Acala 1517 Cotton as shown in Table 2 and also illustrated in figures 8 through 12 as well as the other more mature fibers.

Table 2. A Comparison of the Acetyl Content of Acala 1517 Cotton at Various Times and Temperatures of Presoaking

Temperature °F.	10 min. % Acetyl	30 min. % Acetyl	60 min. % Acetyl
70	2.16	2.78	4.20
100	6.61	6.78	9.75
130	9.80	12.73	15.05
170	14.01	14.45	14.42

As the time of presoaking increased, the effect of the temperature decreased, with the exception of the 70° F. run. This is shown by the fact that at 100° F. the acetyl content reached a maximum in about 120 minutes, at 130° F. in about 60 minutes and at 170° F. in about 30 minutes. This was true for all the cottons with the exception of the immature Memphis Cotton which reached its maximum acetyl content in 10 minutes at the three higher temperatures as shown in Figure 13. After the maximum was attained there were only slight fluctuations in acetyl content with time of presoaking, which may be attributed to small variations in the cotton, experimental error, etc. The maximum degree of acetylation for each variety of fiber was approximately the same for all three of the higher temperatures. This is illustrated in Figures 13 through 18 by noting that the lines representing the three higher presoak temperatures practically coincide once the maximum acetylation has been reached. For example, the acetyl content of the immature Memphis Cotton reaches its maximum acetylation in 10 minutes and then only varies from a low of 16.07% acetyl at the 10 minute 170° F. presoak to a high of 17.99% at the 240 minute 130° F. presoak, a difference of less than 2 per cent while the most mature cotton, Lockett 140, reached its maximum at 60 minutes and varies from a low of 9.04 per cent acetyl content at the 60 minute 100° F. presoak to a high of 10.06 per cent acetyl content at the 240 minute 100° F. presoak, a difference of one per cent.

The maximum acetyl content was never established at 70° F. within the time range studied as shown in Table 3. The acetyl content was still increasing at the end of 240 minutes for all the varieties of cotton and

the degree of acetylation for the various times was considerably below that of the other temperatures. With the exception of the most immature fibers, Memphis and Empire Bale 92, the acetyl content at the end of 240 minutes was only about half the maximum obtained. The same varieties of conditioned cotton were presoaked for 18 hours at 70° F. by Blandin[1] and a comparison of his results with the acetyl values obtained for 240 minutes at 70° F. is shown below.

Table 3. A Comparison of the Acetyl Content of Various Cotton Fibers Presoaked at 70° F. for Different Times

Cotton	240 min. 70° F.	18 hours 70° F.
Memphis	14.44	16.75
Empire Bale 92	7.27	10.75
Bob Shaw	4.42	8.27
Stoneville 2B	8.03	12.23
Acala 1517	6.93	10.97
Lockett 140	4.00	8.56

At the end of 18 hours at 70° F. the acetyl content of the fibers were still slightly below the maximum obtained at the higher presoaking temperatures.

The hot glacial acetic acid apparently enlarges the capillaries of the fibers at a greater rate and the saturation is reached much sooner

[1]Sherman W. Blandin, <u>A Study of the Effect of Scouring and Moisture Preconditioning on the Rate and Degree of Acetylation of Various Varieties of Cotton Fibers</u>. Master's Thesis, Georgia Institute of Technology, 1953.

thus permitting a more rapid diffusion of the acetylating mixture into the inner parts of the fiber. The higher the temperature the faster the saturation point is reached.

The different varieties of cotton reacted at different rates to the effect of the temperature of presoaking. The most immature fiber, Memphis, reached its maximum acetylation in ten minutes at the three higher presoaking temperatures while the more mature fibers, Bob Shaw and Lockett 140, took a considerably longer time, i.e. 30 minutes at 170° F., 60 minutes at 130° F., and 120 minutes at 100° F., to reach maximum acetylation. Generally speaking the more mature the fiber, the less the degree of acetylation under constant conditions. This may be shown by comparison of the acetyl content in Tables 4 through 7 with the maturity values shown in Table I. The order of increasing maturity is (1) Memphis, (2) Empire Bale 92, (3) Stoneville 2B, (4) Acala 1517, (5) Bob Shaw and (6) Lockett 140, while the order of decreasing acetyl content is (1) Memphis, (2) Acala 1517, (3) Stoneville 2B, (4) Empire Bale 92, (5) Bob Shaw, and (6) Lockett 140. Thus with the exception of Empire Bale 92 and Acala 1517 whose order is reversed, the move mature the fiber the less the acetyl content.

The higher degree of acetylation of the immature fibers is due in part to the greater percentage of amorphous structure and less per cent of crystallinity. The immature fibers exhibit greater swelling due to the collapse of the thin fiber walls and thus allowing greater diffusion of the acetylating mixture. Other factors such as gum and wax content, mineral content, alpha cellulose, etc. may also effect the degree of acetylation.

A comparison of the Micronaire Readings in Table I with the acetyl content shows that generally the higher the Micronaire Reading the lower the acetyl content of the cotton. This is to be expected as the more mature the cotton the thicker the fiber walls and thus the higher the Micronaire Reading. Both the fiber fineness and the maturity effect the degree of acetylation and both factors have to be taken into consideration in determining which cotton would be expected to exhibit the highest acetyl content.

The effect of the temperature of acetylation on the degree of acetylation is determined in Part II of this study. The results of these determinations are shown in Table 8 and Figure 7.

An increase of 24° F., from 58° F. to 82° F., doubled the acetyl values obtained for the various fibers. For example, the immature Memphis Cotton increased in acetyl content from 12.69% to 23.68%, the mature Lockett 140 cotton increased from 7.96% to 19.88%, and an intermediate cotton, Stoneville 2B increased from 10.68% to 21.51%. The effect of the temperature of acetylation was determined in six degree increments and the differences in acetyl content between increments was found to be relatively constant. The fiber degradation increased noticeably with each increase in temperature as shown in Table 23 in the appendix. At 82° F. the degradation to the cotton fibers was so great that the tensile strength of the fibers could not be determined.

The moisture content, fiber fineness, and tensile strength of the acetylated cotton samples, Tables 9 through 23, were determined by

Holloway.[2] It was found that as the acetyl values increased the moisture content decreased and the fiber fineness decreased (Micronaire Readings increased). The fiber strength generally decreased with increasing acetyl content and increasing time of presoaking. The tensile strength per unit weight of cellulose however, probably experiences little change except possibly in the 76° F. and 82° F. acetylations as the fiber weight increases approximately 25% at the monoacetate stage.

The dye test for evenness of acetylation produced uniform coloring throughout the cotton samples and thus showed that the acetylation was even throughout the cotton fibers.

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[2] Benjamin Holloway, A Comparison of the Physical Characteristics of Acetylated Cotton with those of Untreated Cottons. Master's Thesis, Georgia Institute of Technology, June 1953.

Table 4. Effect of Time of Presoaking at 70° F. on the Degree of Acetylation*

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	8.06	10.50	13.05	13.74	14.44
Empire Bale 92	1.52	2.75	3.83	5.07	6.25	7.27
Bob Shaw	0.86	1.46	2.35	3.07	4.38	4.42
Stoneville 2B	1.37	3.36	3.97	4.87	6.80	8.03
Acala 1517	1.43	2.16	2.78	4.20	5.61	6.93
Lockett 140	1.02	1.33	1.84	2.48	2.68	4.00

Table 5. Effect of Time of Presoaking at 100° F. on the Degree of Acetylation

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	16.07	16.21	17.20	17.33	17.52
Empire Bale 92	1.52	7.87	9.10	9.86	11.77	12.27
Bob Shaw	0.86	4.38	4.92	6.44	8.45	9.34
Stoneville 2B	1.37	7.90	8.40	11.45	13.78	14.00
Acala 1517	1.43	6.61	6.78	9.75	14.30	15.40
Lockett 140	1.02	4.24	5.10	9.04	9.06	10.06

*All acetylations in Tables 4 through 7 were carried out at 64° F. for 45 minutes.

Table 6. Effect of Time of Presoaking at 130° F. on the Degree of Acetylation

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	16.11	16.53	17.17	17.00	17.99
Empire Bale 92	1.52	8.42	10.35	12.14	12.25	12.99
Bob Shaw	0.86	6.07	8.30	9.87	10.55	11.10
Stoneville 2B	1.37	11.58	12.59	13.18	13.28	14.00
Acala 1517	1.43	9.80	12.73	15.05	15.30	16.24
Lockett 140	1.02	6.20	7.41	9.91	9.82	9.96

Table 7. Effect of Time of Presoaking at 170° F. on the Degree of Acetylation

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	3.72	16.51	16.95	16.51	16.82	16.60
Empire Bale 92	1.52	11.41	11.95	12.08	11.82	12.53
Bob Shaw	0.86	8.89	9.52	9.62	9.95	10.45
Stoneville 2B	1.37	12.79	12.81	13.25	12.71	13.88
Acala 1517	1.43	14.01	14.45	14.42	14.96	15.36
Lockett 140	1.02	8.86	9.27	9.81	9.75	9.63

Table 8. Effect of Temperature of Acetylation on the Degree of Acetylation*

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.
Memphis	12.69	16.51	19.22	20.71	23.68
Empire Bale 92	9.12	11.41	13.51	17.10	20.36
Bob Shaw	7.48	8.89	10.68	15.30	19.28
Stoneville 2B	10.68	12.79	15.40	18.31	21.51
Acala 1517	10.76	14.01	16.77	20.03	23.12
Lockett 140	7.96	8.86	10.98	14.72	19.88

*Presoaked for 10 minutes at 170° F. and acetylated for 45 minutes.

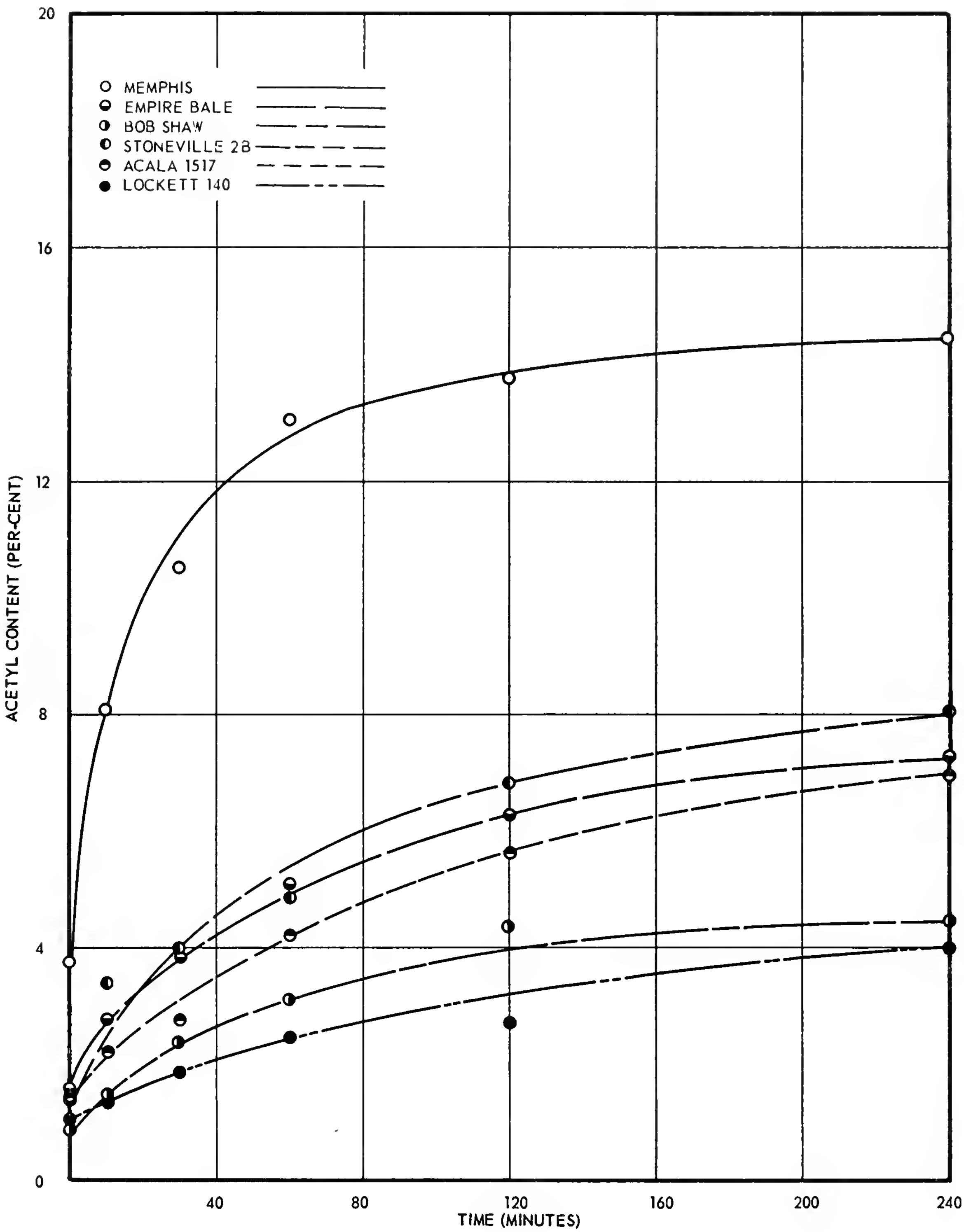

Figure 3. The Change in Acetyl Content with Presoaking Time at 70° F.

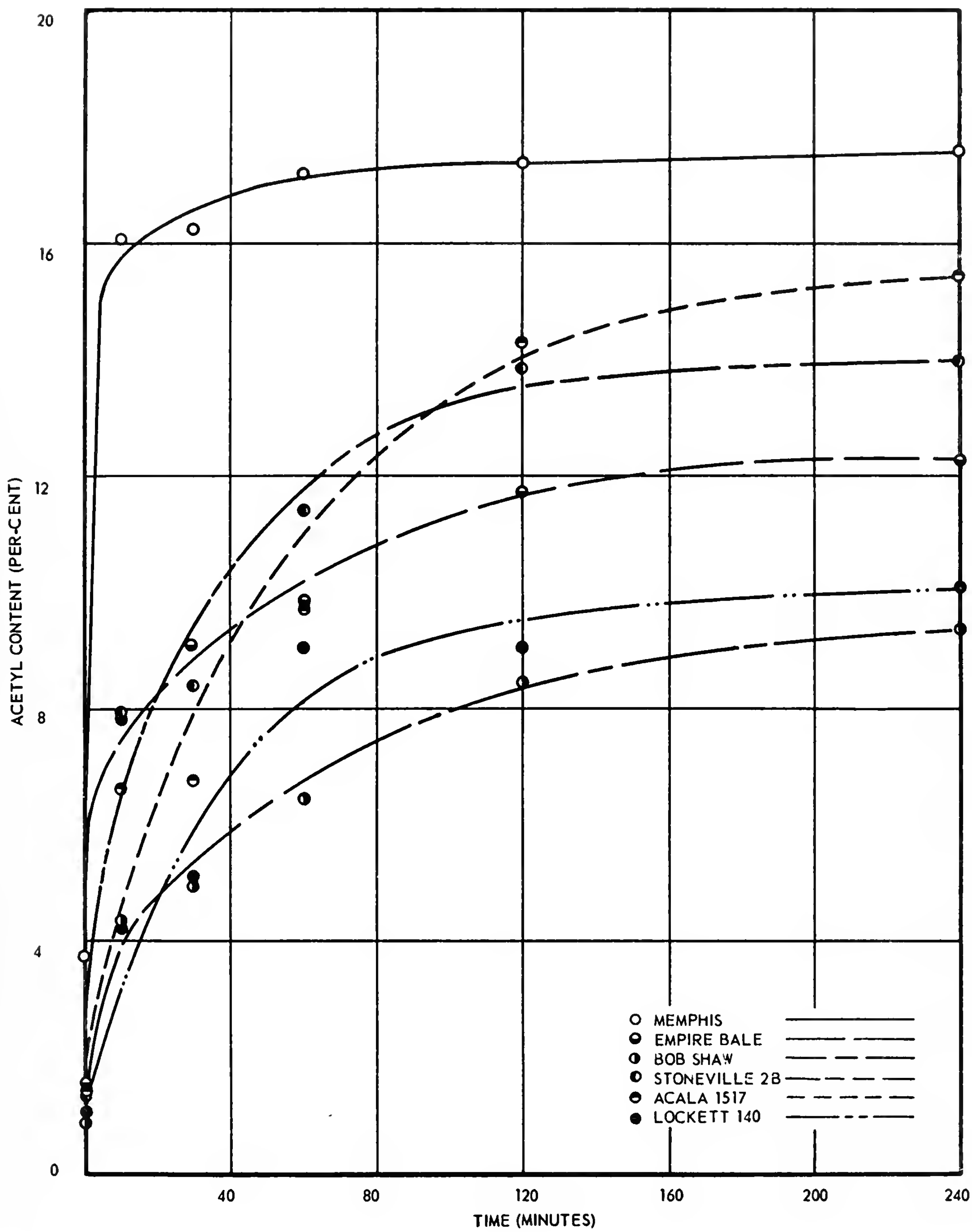

Figure 4. The Change in Acetyl Content with Presoaking Time at 100° F.

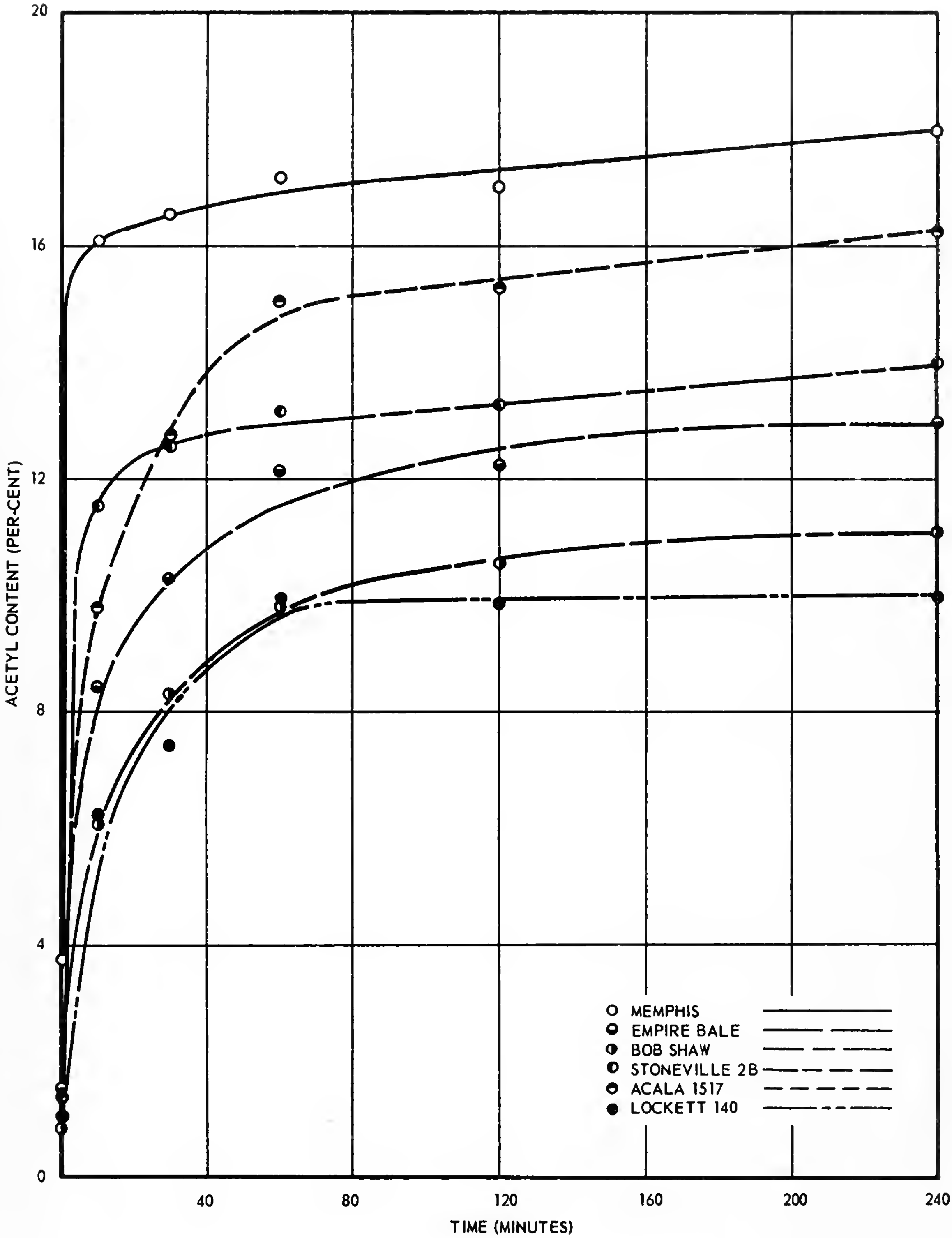

Figure 5. The Change in Acetyl Content with Presoaking Time at 130 F.

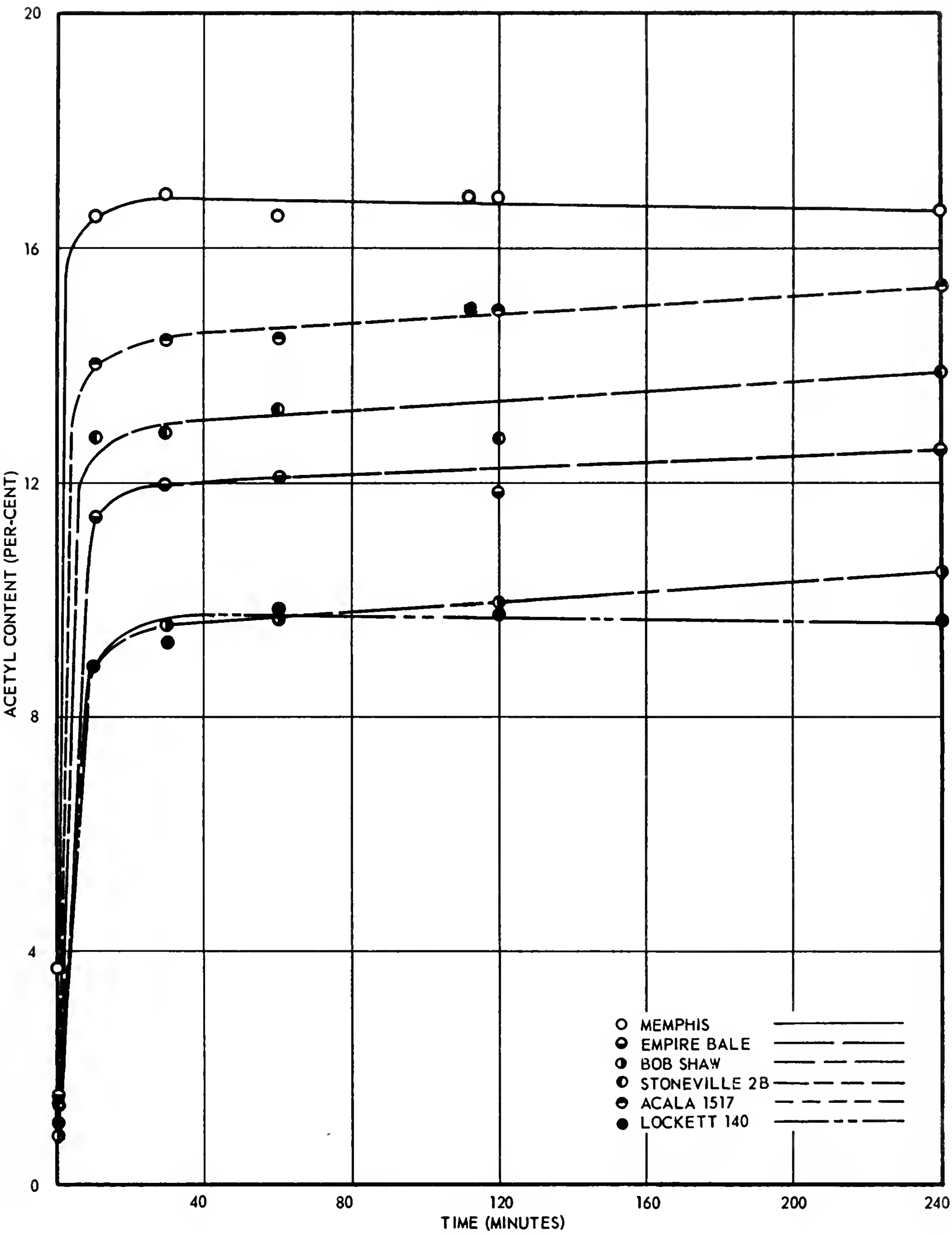

Figure 6. The Change in Acetyl Content with Presoaking Time at 170 F.

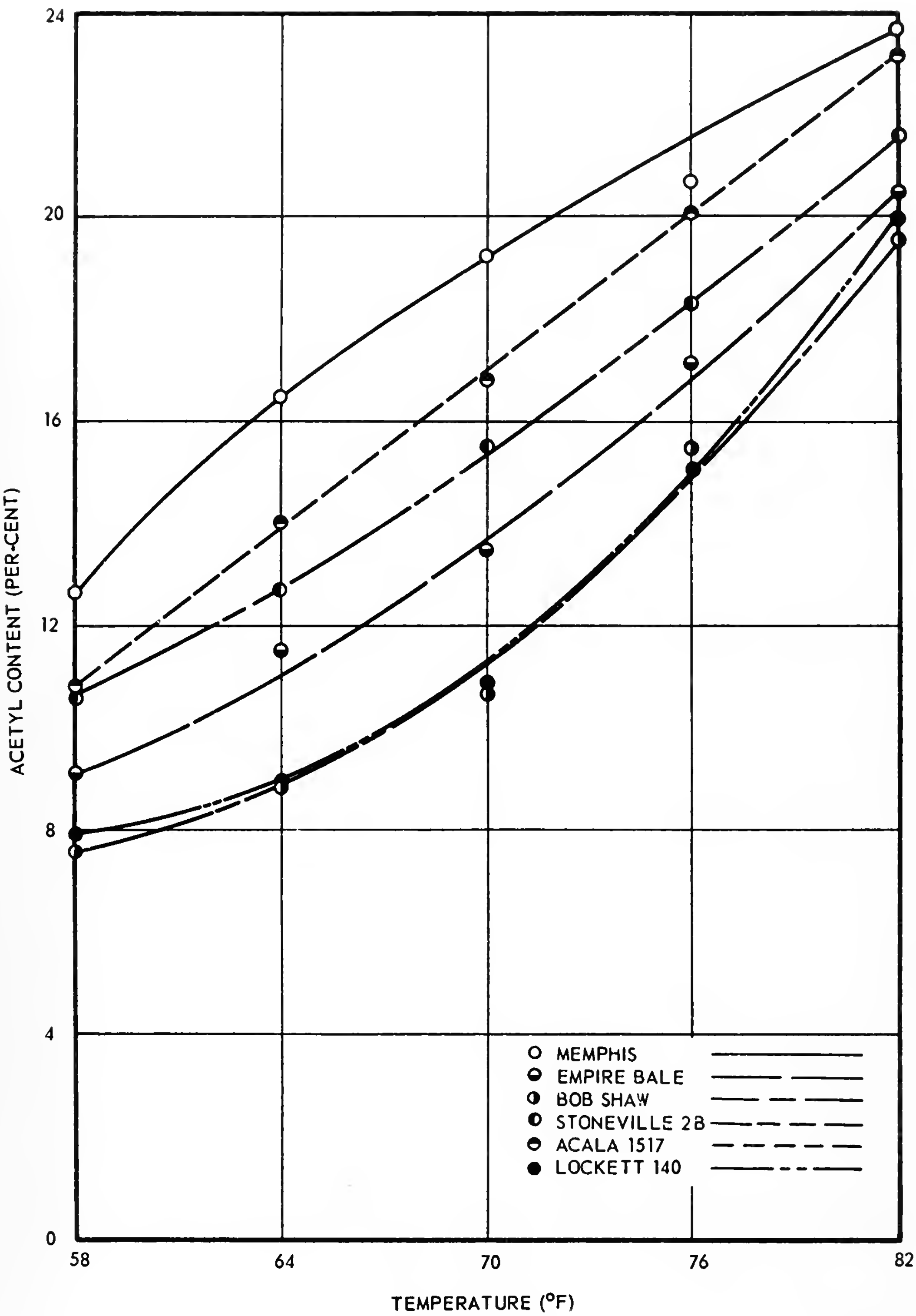

Figure 7. The Change in Acetyl Content with Acetylation Temperature using a 10 minute, 170 F. Presoak.

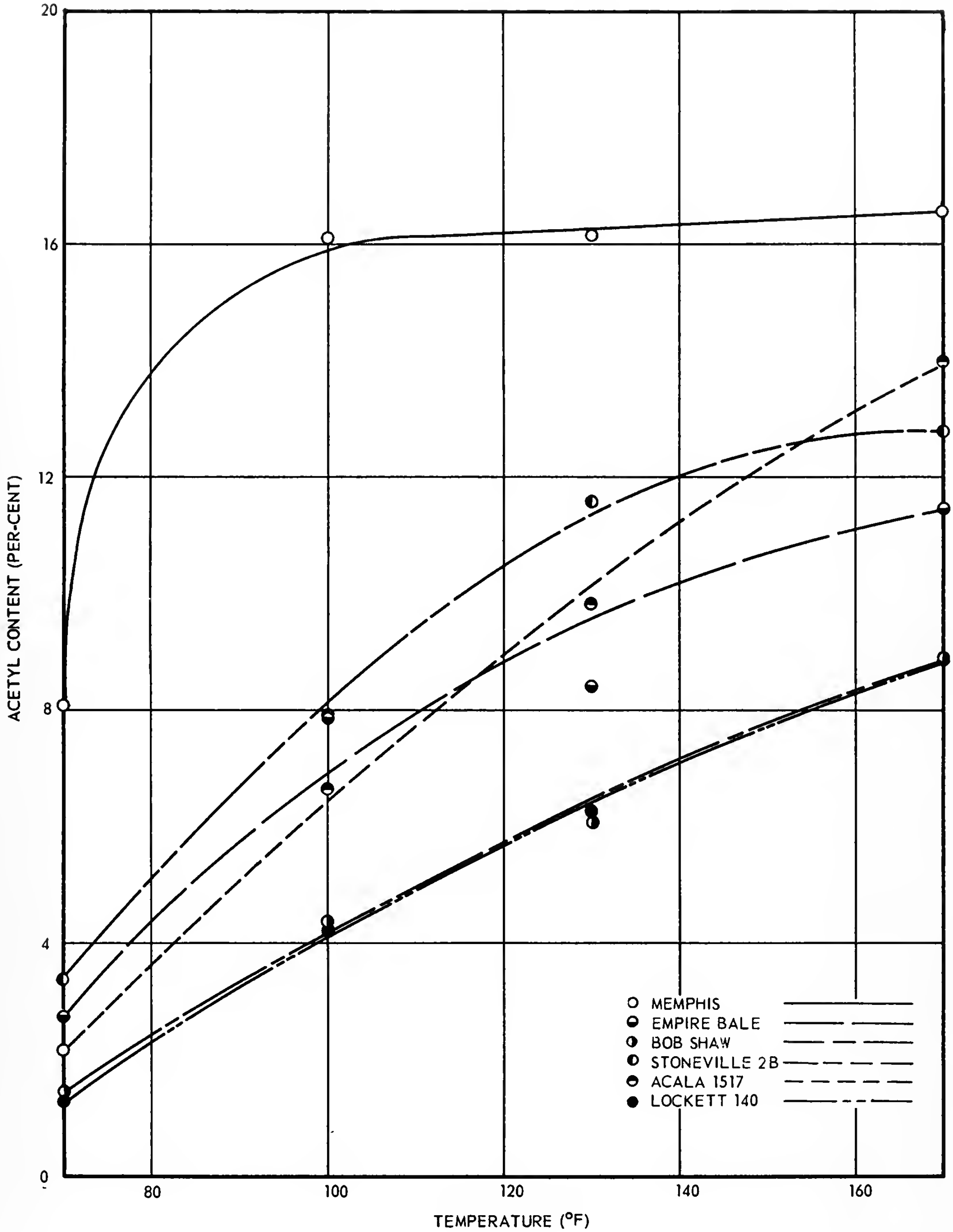

Figure 8. The Change in Acetyl Content with Presoaking Temperature using a 10 Minute Presoaking Period.

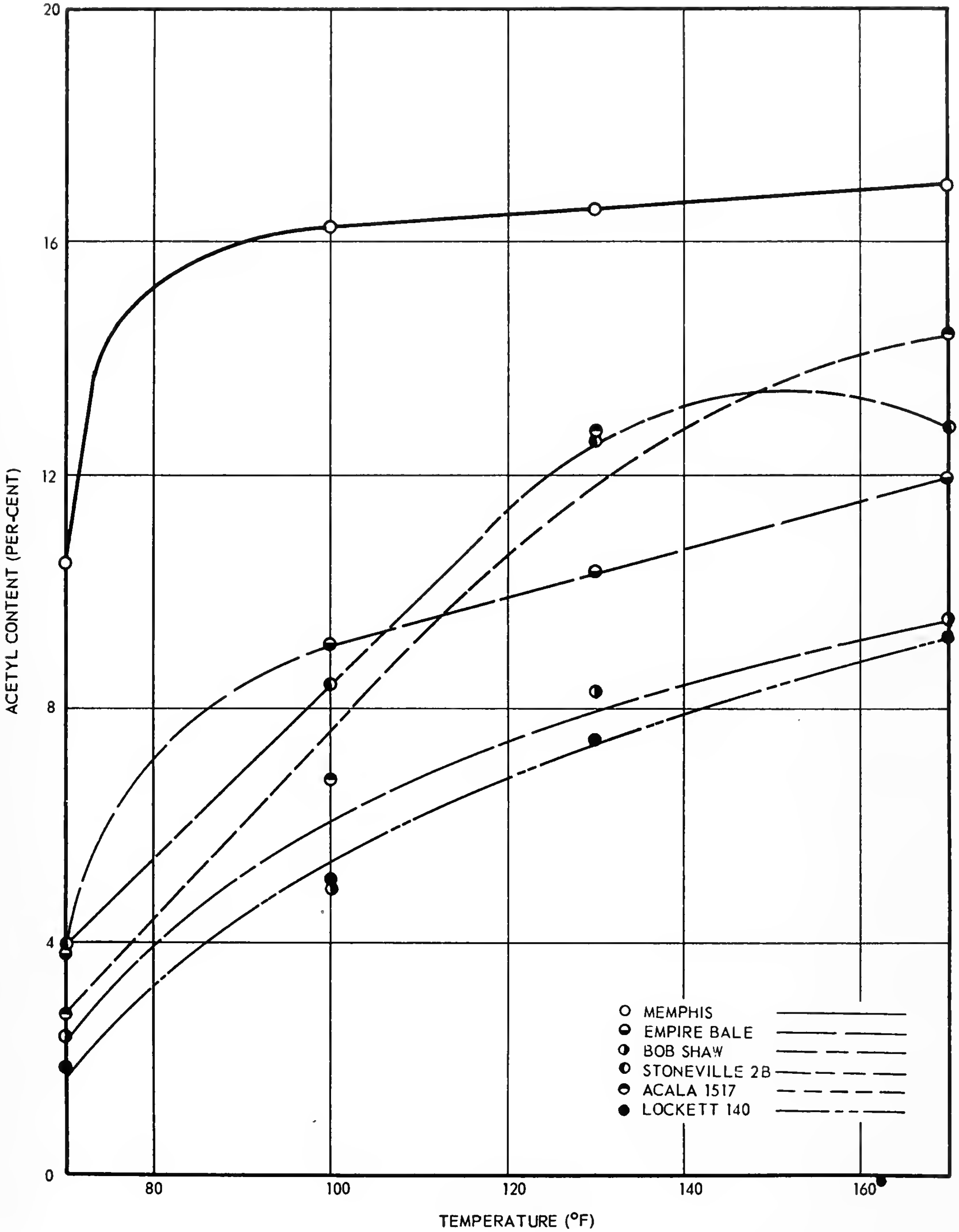

Figure 9. The Change in Acetyl Content with Presoaking Temperature using a 30 minute Presoaking Period.

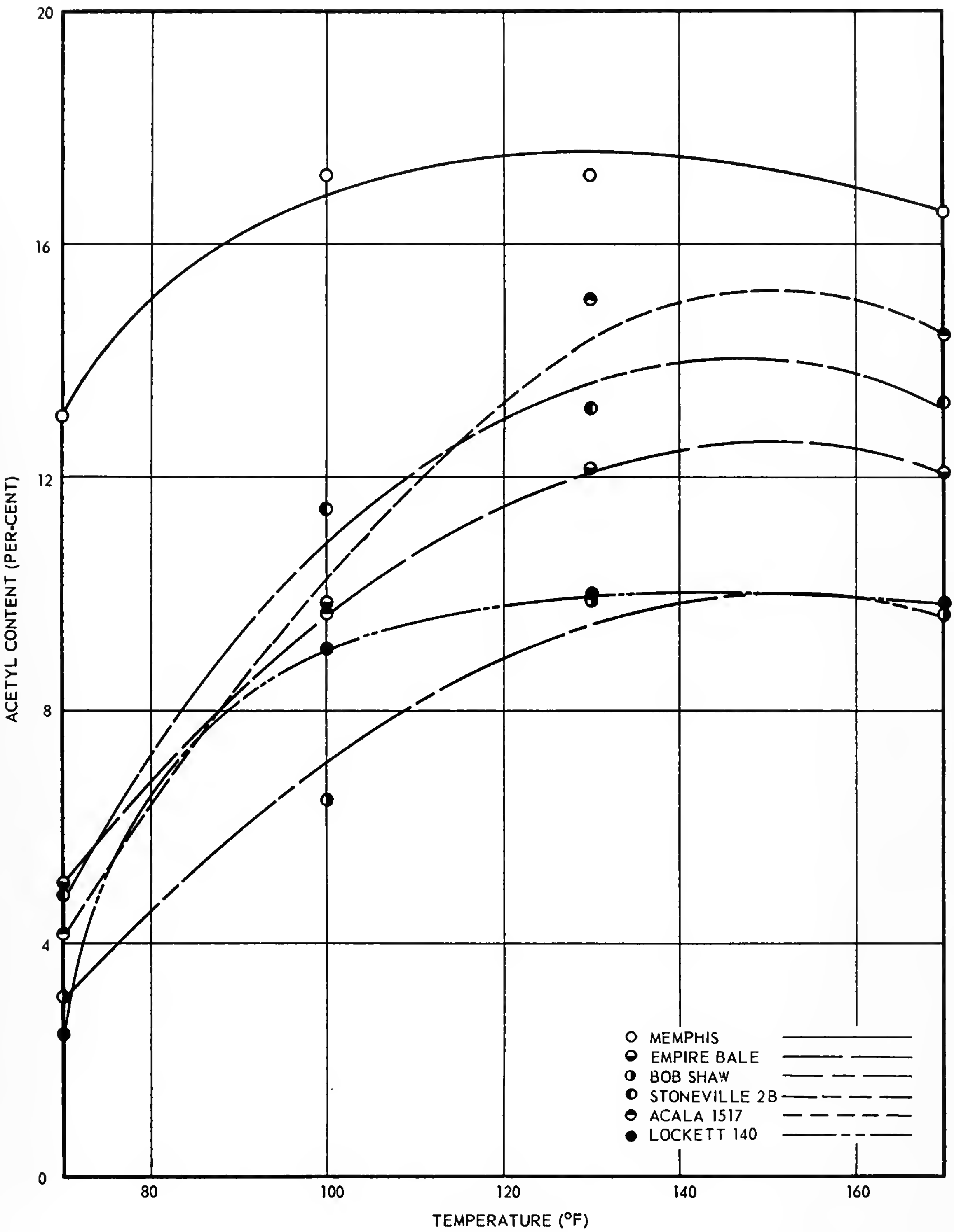

Figure 10. The Change in Acetyl Content with Presoaking Temperature using a 60 Minute Presoaking Period.

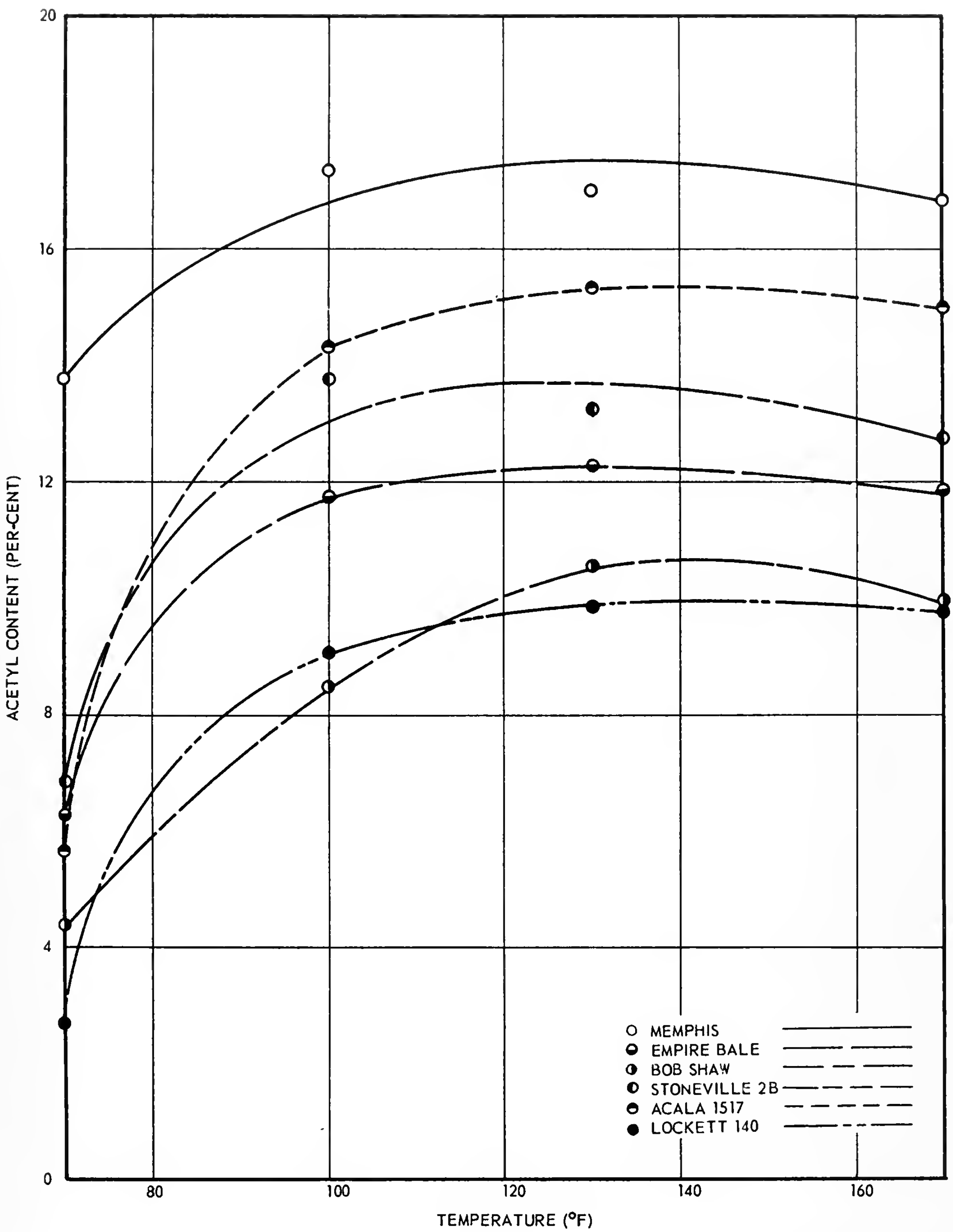

Figure 11. The Change in Acetyl Content with Presoaking Temperature using a 120 Minute Presoaking Period.

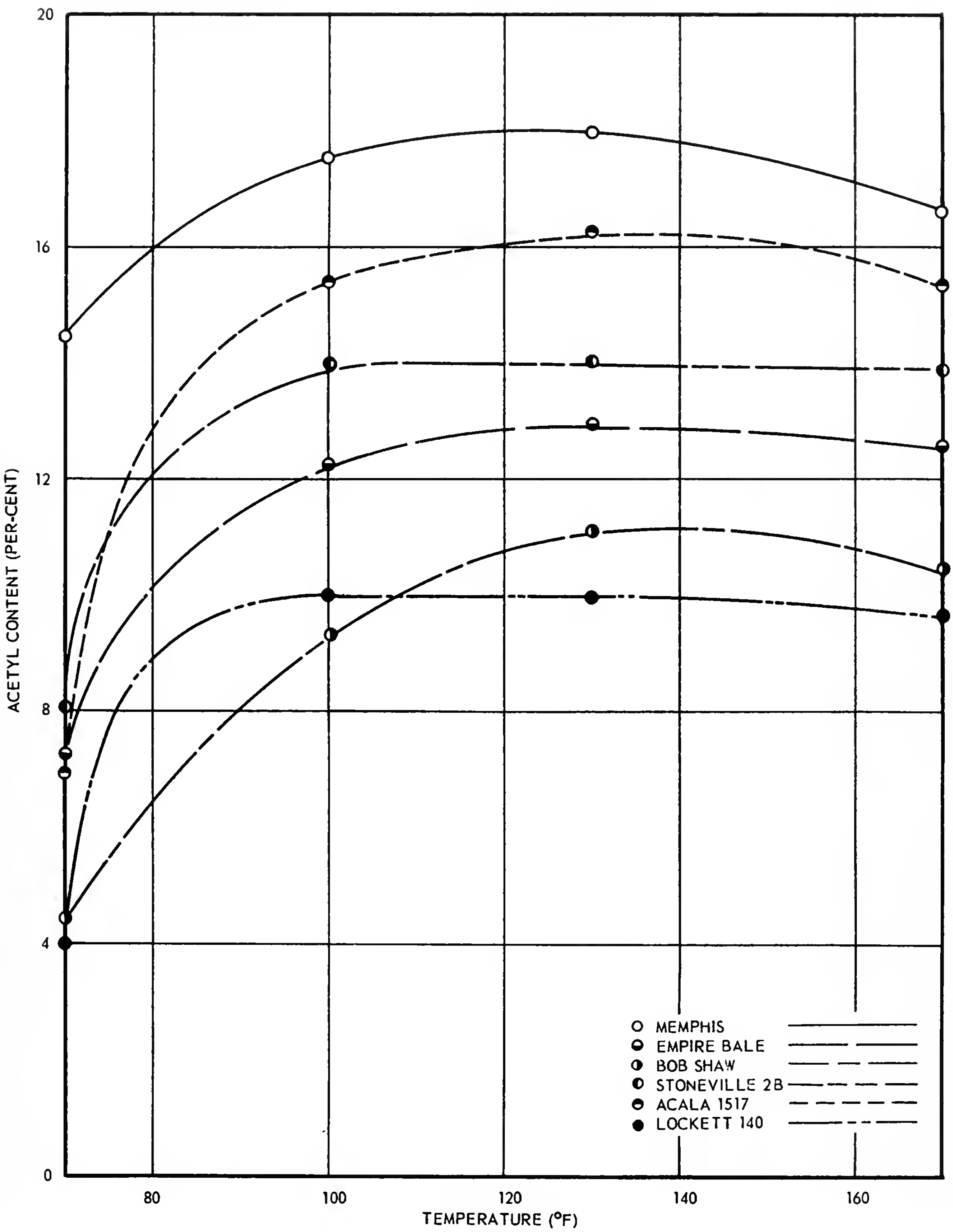

Figure 12. The Change in Acetyl Content with Presoaking Temperature using a 240 minute Presoaking Period.

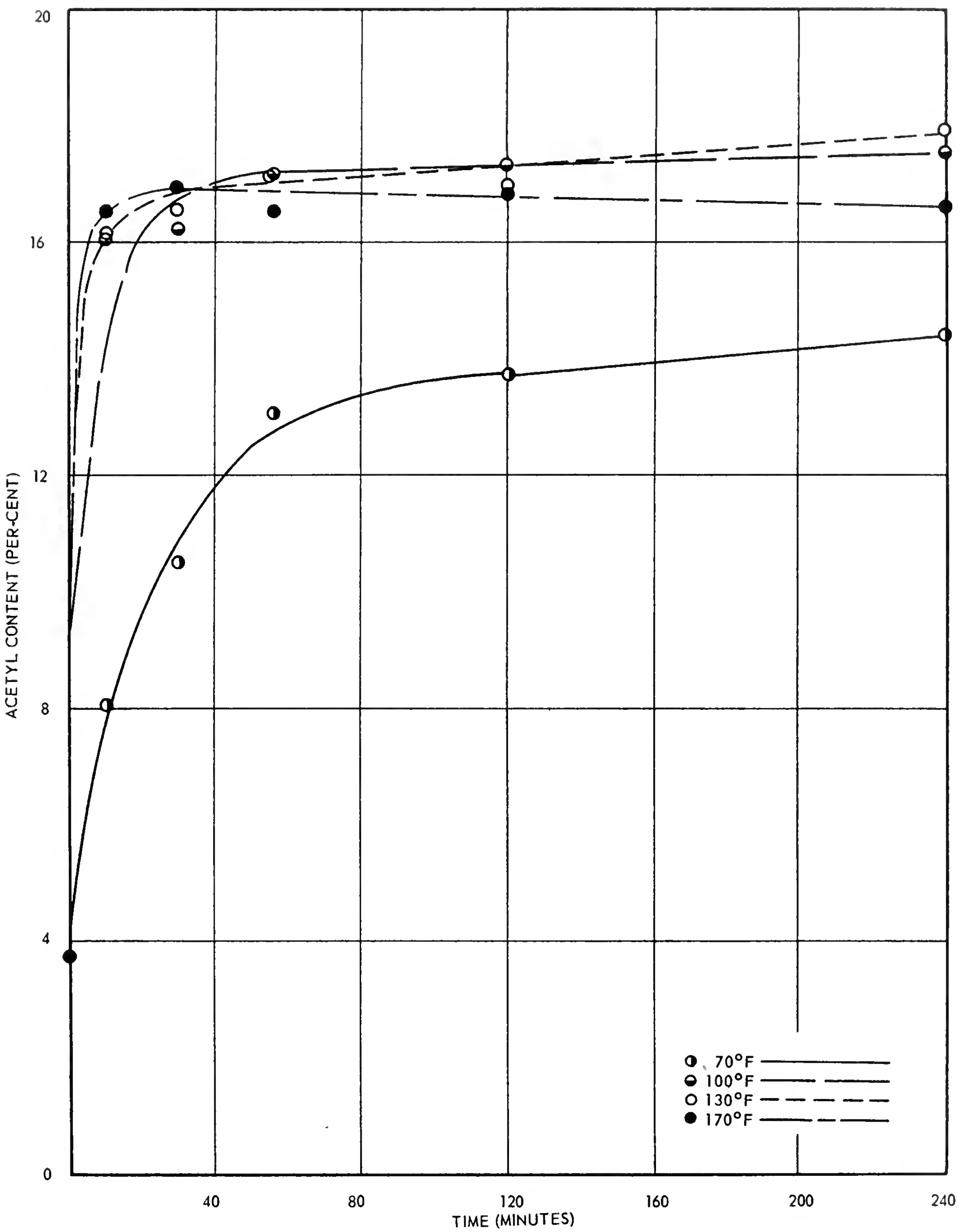

Figure 13. The Change in Acetyl Content with Presoaking Time for Memphis Cotton at Different Presoak Temperatures.

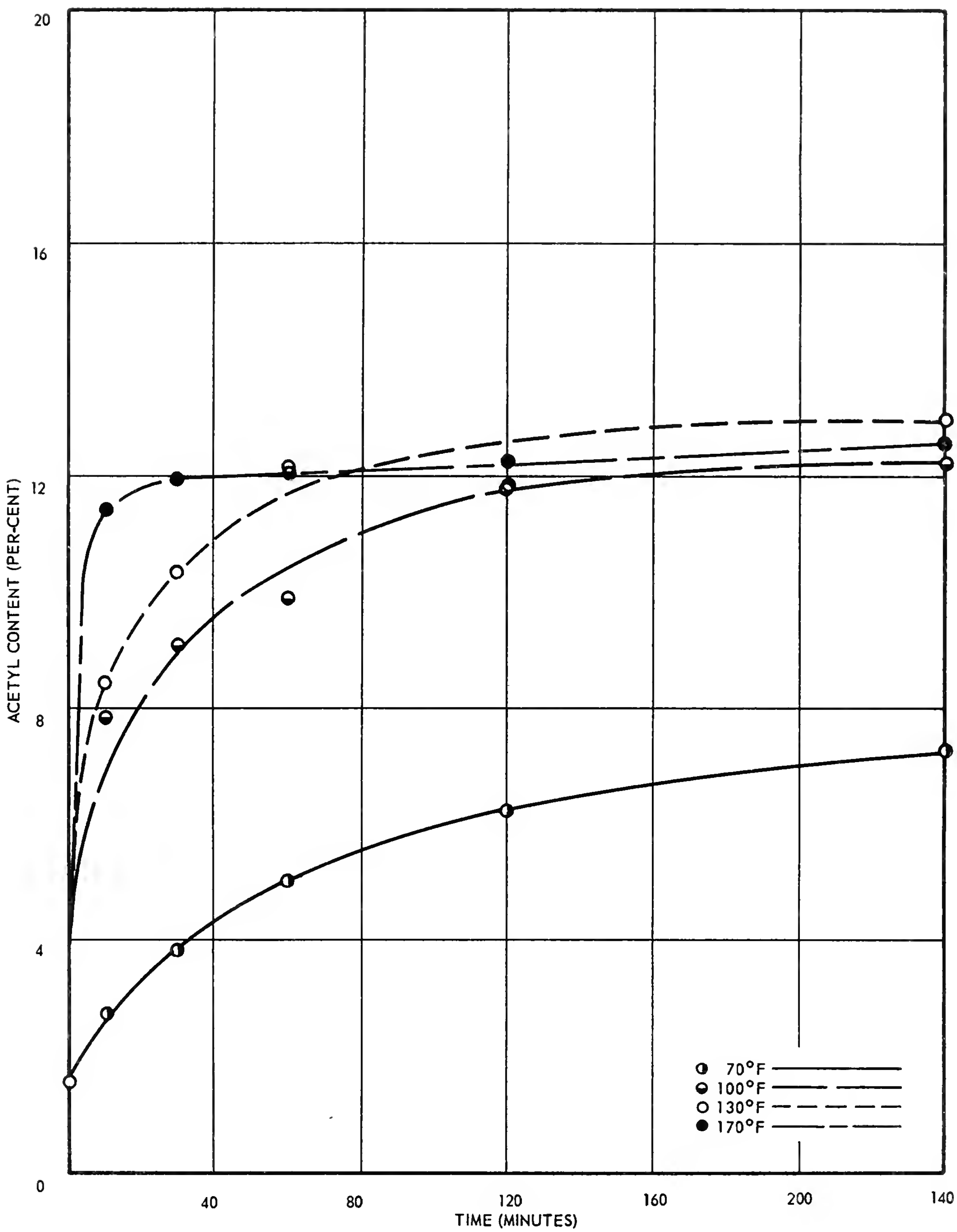

Figure 14. The Change in Acetyl Content with Presoaking Time for Empire Bale 92 Cotton at Different Presoak Temperatures.

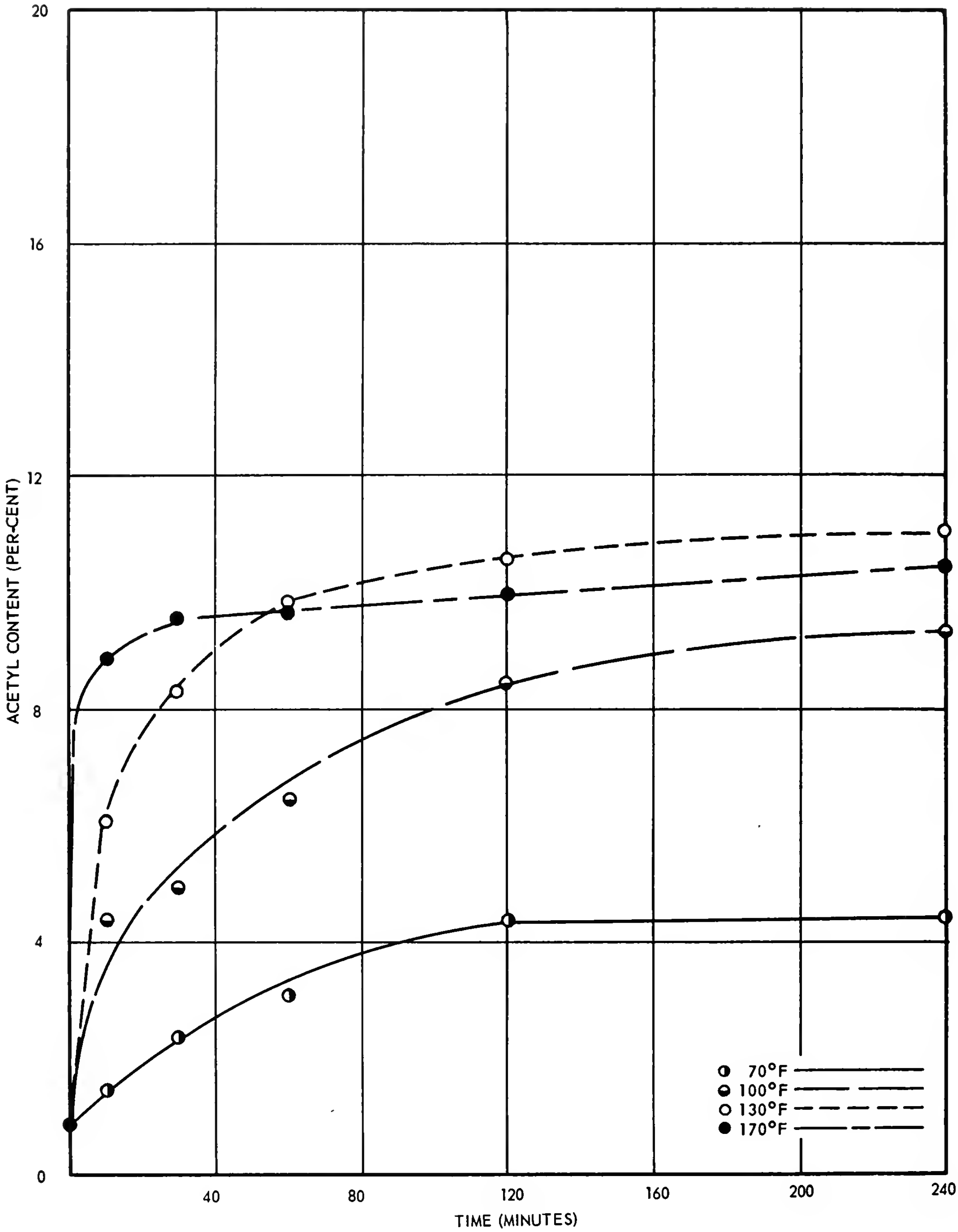

Figure 15. The Change in Acetyl Content with Presoaking Time for Bob Shaw Cotton at Different Presoak Temperatures.

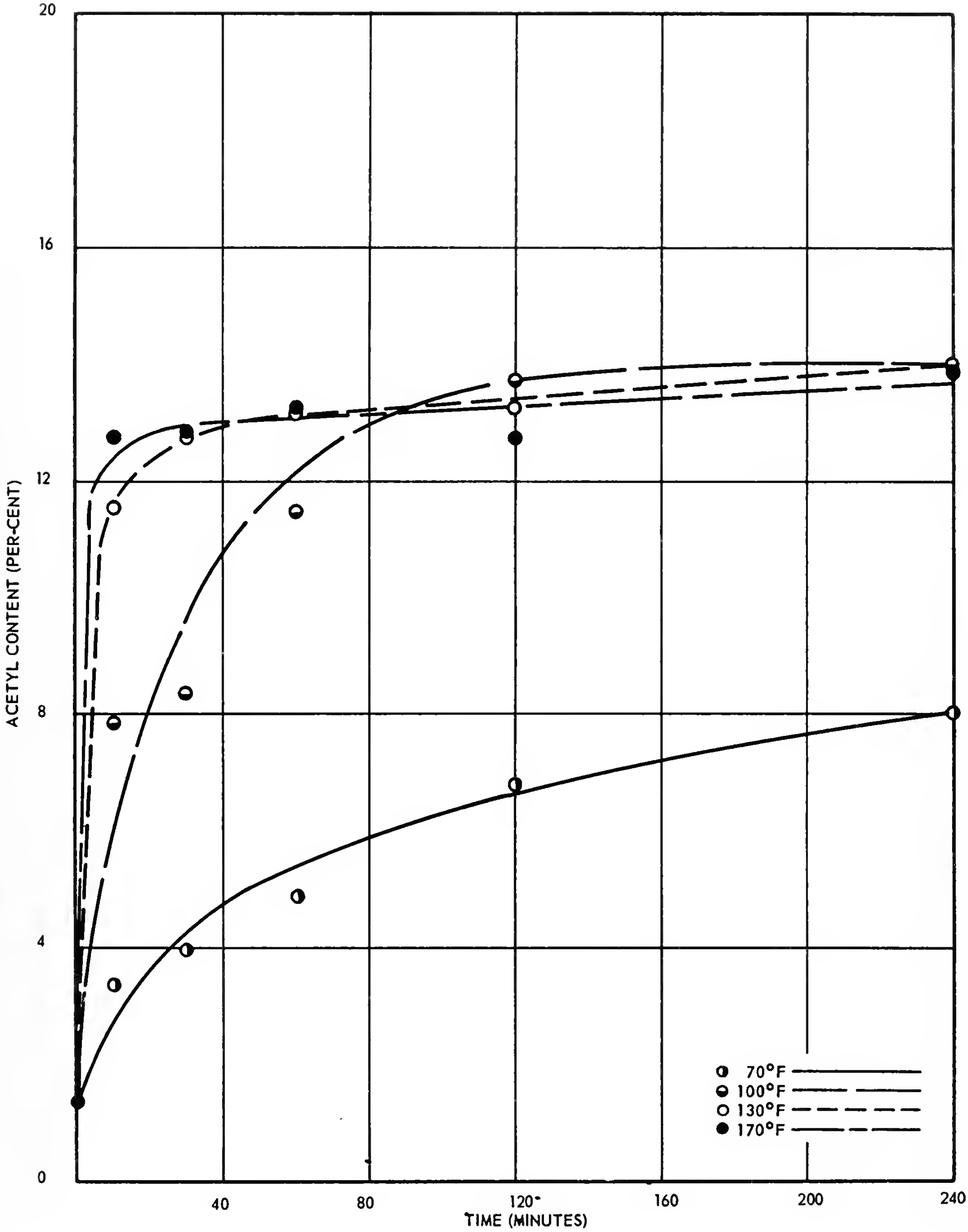

Figure 16. The Change in Acetyl Content with Presoaking Time for Stoneville 2 B Cotton at Different Presoak Temperatures.

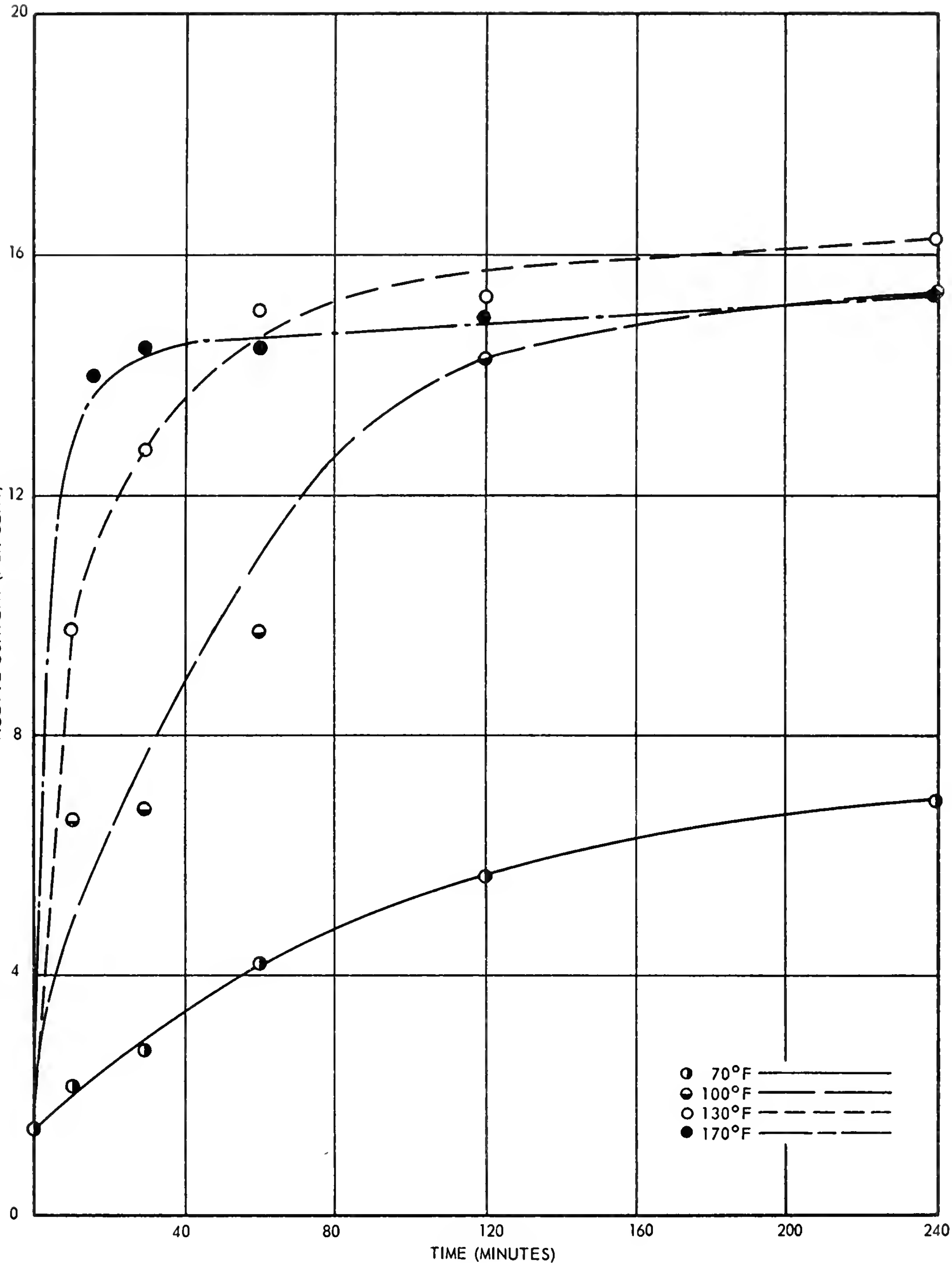

Figure 17. The Change in Acetyl Content with Presoaking Time for Acala 1517 Cotton at Different Presoak Temperatures.

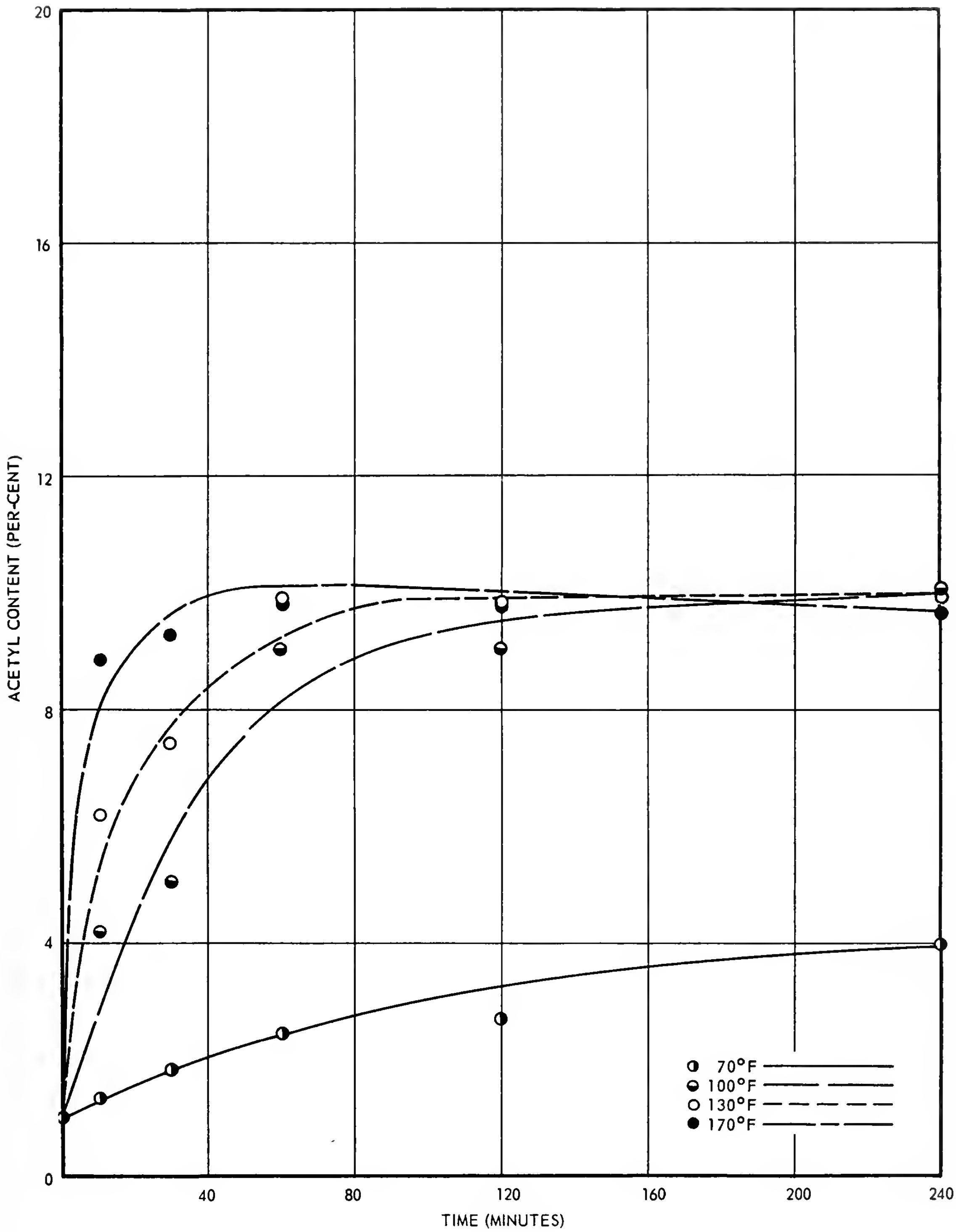

Figure 18. The Change in Acetyl Content with Presoaking Time for Lockett 140 Cotton at Different Presoak Temperatures.

CHAPTER VI

CONCLUSIONS

An increase in the temperature of the glacial acetic acid used for presoaking increases the rate in which maximum acetylation is reached under constant acetylation conditions.

Higher degrees of acetylation are obtained at the higher presoaking temperatures for short periods, 10 to 60 minutes, of presoaking.

Increasing the temperature of the presoaking acid increases the rate of acetylation but it does not effect the maximum degree of acetylation obtained under constant acetylation conditions.

The optimum time and temperature of presoaking (maximum acetylation in the shortest presoaking time) was found to be 10 minutes at 170° F.

Different fibers acetylated at different rates and degrees under constant conditions of presoaking and acetylation.

The order of maximum acetyl content under the same acetylating conditions obtained for the various fibers was Memphis, Acala 1517, Stoneville 2B, Empire Bale, Bob Shaw, and Lockett 140.

Generally the more immature the fiber the greater the rate and degree of acetylation under constant conditions of presoaking and acetylation and conversely the more mature the fiber the less the rate and degree of acetylation under constant conditions.

The higher the temperature of acetylation, the greater the degree of acetylation and the greater the degradation of the cotton fibers.

In general, under the same conditions of presoaking and time of acetylation, an increase of 24° F., from 58° F. to 82° F., approximately doubled the acetyl content obtained but weakened the fibers so that fiber strengths were unobtainable at 82° F.

The method of presoaking and acetylation used in this study produced evenly acetylated cotton fibers.

CHAPTER VII

RECOMMENDATIONS

It is recommended that further research be conducted into the effects of presoaking above 130° F. using different times and temperatures of acetylation. The conditions of time and temperature required for obtaining the desired acetyl content with the least amount of degradation could thus be determined.

The possibility of producing acetylated cotton on a continuous range by first scouring the raw stock followed by a short high temperature presoak and short acetylation should be investigated in order to insure uniformity in degree of acetylation of the various fibers, less degradation of the fibers and shorter overall time of production.

The use of high temperature presoaking in partially acetylating yarns and fabrics should be investigated.

The effect of other chemical and physical properties of the fibers, such as alpha cellulose, gum and wax content, ash content, and crystallinity, on the partial acetylation of cotton should be investigated.

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APPENDIX

Table 9. Per Cent Moisture Content of Acetylated Samples Presoaked at 70° F.*

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	5.8	5.6	5.4	4.8	4.4
Empire Bale 92	5.4	5.7	5.7	5.6	5.4	5.1
Bob Shaw	5.4	5.5	5.5	5.4	5.3	4.9
Stoneville 2B	5.7	5.8	5.7	5.6	5.5	5.0
Acala 1517	5.7	5.8	5.8	5.9	5.3	5.3
Lockett 140	5.8	5.9	5.9	5.9	5.7	5.5

Table 10. Per Cent Moisture Content of Acetylated Samples Presoaked at 100° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	4.9	4.7	4.4	4.0	4.0
Empire Bale 92	5.4	5.3	4.9	5.1	4.6	4.8
Bob Shaw	5.4	5.0	5.3	4.9	5.2	4.9
Stoneville 2B	5.7	5.4	5.1	5.0	4.2	3.9
Acala 1517	5.7	5.8	5.6	5.3	4.4	3.8
Lockett 140	5.8	5.2	5.3	5.5	5.1	4.4

*All acetylations in Tables 7-10, 12-15, and 17-20 were 45 minutes at 64° F.

Table 11. Per Cent Moisture Content of Acetylated Samples Presoaked at 130° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	4.6	4.1	3.7	3.8	3.9
Empire Bale 92	5.4	4.8	4.7	4.4	4.0	4.2
Bob Shaw	5.4	5.2	4.9	4.5	4.4	4.3
Stoneville	5.7	4.8	4.5	4.2	4.2	4.1
Acala 1517	5.7	4.3	4.5	4.3	3.9	4.2
Lockett 140	5.8	5.2	5.0	4.5	4.5	4.4

Table 12. Per Cent Moisture Content of Acetylated Samples Presoaked at 170° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	5.8	4.7	4.7	4.3	4.2	4.3
Empire Bale 92	5.4	5.2	5.1	4.6	4.9	4.5
Bob Shaw	5.4	5.4	5.0	4.9	4.2	4.4
Stoneville 2B	5.7	4.8	5.3	5.2	4.5	4.3
Acala 1517	5.7	5.0	5.3	5.0	4.6	4.3
Lockett 140	5.8	5.2	5.3	5.3	4.8	4.6

Table 13. Per Cent Moisture Content of Samples Acetylated at Different Temperatures*

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.
Memphis	4.9	4.7	4.0	4.2	3.9
Empire Bale 92	4.9	5.2	4.2	4.4	4.0
Bob Shaw	5.0	5.4	4.6	3.9	3.5
Stoneville 2B	5.3	4.8	4.3	4.2	3.7
Acala 1517	5.3	5.0	4.3	4.0	3.4
Lockett 140	5.4	5.2	4.3	4.4	3.7

*Acetylations in Tables 11, 16, 21, carried out for 45 minutes with a presoak of 170° F. for 10 minutes.

Table 14. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 70° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.62	2.63	2.68	2.68	2.70
Empire Bale 92	3.95	3.87	4.00	3.90	3.90	3.97
Bob Shaw	5.43	5.30	5.37	5.32	5.35	5.40
Stoneville 2B	3.80	3.66	3.83	3.75	3.73	3.83
Acala 1517	4.27	4.20	4.30	4.22	4.25	4.28
Lockett 140	6.03	5.92	6.05	5.95	6.05	6.03

Table 15. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 100° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.80	2.70	2.82	2.75	2.75
Empire Bale 92	3.95	3.90	3.78	4.10	3.87	3.92
Bob Shaw	5.43	5.35	5.07	5.35	5.27	5.43
Stoneville 2B	3.80	3.80	3.70	3.88	3.73	3.83
Acala 1517	4.27	4.30	4.17	4.30	4.23	4.35
Lockett 140	6.03	5.90	5.95	5.95	5.93	5.96

Table 16. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 130° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.85	2.85	2.87	2.80	2.87
Empire Bale 92	3.95	4.02	4.00	4.13	3.95	4.01
Bob Shaw	5.43	5.47	5.40	5.50	5.22	5.42
Stoneville 2B	3.80	3.90	3.91	3.93	3.80	3.93
Acala 1517	4.27	4.35	4.33	4.45	4.25	4.30
Lockett 140	6.03	5.97	6.00	6.08	6.02	6.18

Table 17. Micronaire Readings (Fiber Fineness) of Acetylated Samples Presoaked at 170° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	2.60	2.63	2.73	2.78	2.73	2.80
Empire Bale 92	3.95	3.93	4.08	4.00	4.10	4.17
Bob Shaw	5.43	5.38	5.40	5.43	5.45	5.40
Stoneville 2B	3.80	3.80	4.02	3.85	4.00	4.10
Acala 1517	4.27	4.22	4.42	4.35	4.35	4.52
Lockett 140	6.03	5.90	6.02	5.97	6.07	6.10

Table 18. Micronaire Readings (Fiber Fineness) of Samples Acetylated at Different Temperatures

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.
Memphis	2.68	2.63	3.00	3.08	3.18
Empire Bale 92	4.02	3.93	4.33	4.28	4.50
Bob Shaw	5.37	5.38	5.73	5.57	5.87
Stoneville 2B	3.65	3.80	4.08	4.10	4.23
Acala 1517	4.32	4.22	4.58	4.60	4.78
Lockett 140	6.00	5.90	6.27	6.10	6.18

Table 19. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 70° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.87	6.68	6.44	6.20	6.97
Empire Bale 92	6.99	7.16	6.92	6.69	6.64	6.51
Bob Shaw	8.11	8.20	8.13	7.93	8.07	8.02
Stoneville 2B	7.71	8.16	7.87	7.96	7.73	7.54
Acala 1517	8.63	8.32	8.11	8.11	8.04	8.01
Lockett 140	7.05	7.32	7.20	7.21	6.98	6.84

Table 20. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 100° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.84	6.37	6.10	5.91	5.70
Empire Bale 92	6.99	6.55	6.80	6.52	6.13	5.98
Bob Shaw	8.11	8.33	8.42	8.07	7.68	7.18
Stoneville 2B	7.71	7.13	7.09	6.93	6.68	5.99
Acala 1517	8.63	8.27	8.50	7.84	7.32	7.28
Lockett 140	7.05	7.43	6.96	6.99	6.85	6.48

Table 21. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 130° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.86	6.78	6.47	6.33	6.05
Empire Bale 92	6.99	6.90	6.39	6.38	6.27	5.91
Bob Shaw	8.11	8.08	7.82	7.74	7.01	6.87
Stoneville 2B	7.71	7.33	7.00	6.95	6.91	6.71
Acala 1517	8.63	8.19	7.64	7.58	7.14	7.04
Lockett 140	7.05	7.50	6.97	6.69	6.70	6.76

Table 22. Pressley Index (Fiber Strength) of Acetylated Samples Presoaked at 170° F.

Cotton	0 Minutes	10 Minutes	30 Minutes	60 Minutes	120 Minutes	240 Minutes
Memphis	6.24	6.89	6.38	6.16	5.97	5.49
Empire Bale 92	6.99	6.91	6.53	6.12	6.05	5.76
Bob Shaw	8.11	7.68	6.95	6.54	6.13	5.61
Stoneville 2B	7.71	6.64	6.21	5.72	5.98	5.88
Acala 1517	8.63	8.29	7.64	6.35	6.35	6.14
Lockett 140	7.05	7.25	6.56	5.97	5.83	5.40

Table 23. Pressley Index (Fiber Strength) of Samples Acetylated at Different Temperatures

Cotton	58° F.	64° F.	70° F.	76° F.	82° F.*
Memphis	6.02	6.89	6.09	5.52	-
Empire Bale 92	6.62	6.91	6.49	6.16	-
Bob Shaw	7.70	6.68	6.84	6.33	-
Stoneville 2B	7.16	6.64	6.80	6.17	-
Acala 1517	8.01	8.29	6.93	6.83	-
Lockett 140	6.53	7.25	6.27	6.01	-

*The fibers were too weak to obtain a satisfactory break.